Detrital zircon U–Pb, Hf isotopes, detrital rutile and whole-rock geochemistry of the Huade Group on the northern margin of the North China Craton: Implications on the breakup of the Columbia supercontinent

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Abstract

Supracrustal successions in the Zhaertai–Bayan Obo–Huade rift zone on the northern margin of the North China Craton include the Zhaertai, Bayan Obo and Huade Groups from west to east. The Huade Group, which has been subdivided into four formations, is composed of basal pebbled quartzites, quartzites, schists, phyllites, marbles, slates and diopsidites. Geochemistry of the metasedimentary succession indicates that source rocks of the lower two formations are felsic, displaying passive margin signatures, whereas those of the upper two formations have continental island arc or active continental margin signatures. U–Pb ages of detrital zircons from the group yielded three age populations of 2690–2450, 2150–1710 and 1660–1330 Ma, of which the youngest age population only exist in the upper Huade Group. The dominant 2690–2450 detrital zircons were likely sourced from the granitic rocks in the Yinshan Block. The subordinate 2150–1710 Ma detrital zircons were probably recycled from the metasedimentary units from the Khondalite Belt and/or derived from the similar aged granitic plutons of the Honggijingzi “Group”, which are also evidenced by the similar geochemical features of rutiles from the Huade Group and the Khondalite Belt. Minor amounts of 1660–1330 Ma detrital zircons may have derived from the North American and/or Baltica cratons connected to the northern margin of the North China Craton in the Columbia supercontinent. The youngest detrital zircon age peak of ∼1337 Ma in the upper Huade Group suggests that the sedimentation began after ∼1.34 Ga. In combination with the ∼1.32 Ga granitoids cross-cutting it, depositional ages of the upper Huade Group can be constrained between ∼1.34 and ∼1.32 Ga. Taking into account the lithostratigraphic features, provenances and depositional ages, a continental rift basin deposit represented by the upper Huade Group on the northern margin of the North China Craton developed between ∼1.34 and ∼1.32 Ga, which indicates that the final breakup of the North China Craton from the Columbia supercontinent happened in the middle Mesoproterozoic.

1. Introduction

Assembly, configuration, accretion and breakup history of a Paleo–Mesoproterozoic supercontinent, referred to as Nuna (Hoffman, 1997) or Columbia (Rogers and Santosh, 2002; Zhao et al., 2002) have been topics of great interest in the last decades (Dalziel, 1991; Hoffman, 1991, 1997; Moores, 1991; Condie, 2002; Rogers and Santosh, 2002; Zhao et al., 2002; Kröner and Cordani, 2003; Zhai and Liu, 2003; Kusky et al., 2007; Ernst et al., 2008; Hou et al., 2008; Li et al., 2008; Reddy and Evans, 2009). Formation of the Columbia supercontinent was thought to have been completed by global-scale 2.0–1.8 Ga collisional events (Zhao et al., 2002, 2003, 2004, 2006). Fragmentation of Columbia commenced at ca. 1.5 Ga with the final breakup at ca. 1.3–1.2 Ga, marked by widespread Mesoproterozoic continental rifting, anorogenic magmatic activity and mafic dike swarms in most of the cratonic blocks forming the supercontinent (Ernst and Buchan, 2001; Zhao et al., 2006; Ernst et al., 2008; Hou et al., 2008; Goldberg, 2010).

In the North China Craton (NCC), the history of assembly, accretion and breakup of the Columbia supercontinent were recorded by three Paleooproterozoic collisional belts (the Khondalite Belt, the Jiao-Liao-Ji Belt and the Trans-North China Orogen),
the 1.78–1.45 Ga Xiong’er volcanic belt and the Mesoproterozoic Zhaertai–Bayan Obo–Huade and Yan-Liao rift zones, respectively (Zhao et al., 2011 and references therein). However, little consensus has been reached regarding the role of the NCC in the Columbia supercontinent because of several unresolved issues. One of these issues concerns when the NCC was involved in the breakup of the Columbia. Some researchers suggested that the continental rifting in the northern NCC had finished before 1.6 Ga and the NCC did not participate in the final breakup of the Columbia at 1.35–1.20 Ga (Rogers and Santosh, 2002; Zhai, 2004; Hou et al., 2008). However, other researchers believed that the 1.33–1.31 Ga rift-related mafic sills and granitoids in the northern NCC led to the final breakup of the Columbia supercontinent (Zhang et al., 2009, 2012a; Shi et al., 2012). Also at issue is the position of the NCC in the Columbia supercontinent. Suggested adjacent craton to the NCC ranges from the Baltica (Rogers and Santosh, 2009), through the India (Zhao et al., 2002, 2004), to the North America (Hou et al., 2008). However, previous configurations of Columbia were mainly based on limited paleomagnetic data or the general geological similarity of the NCC with those of other cratonic blocks, few investigations have been carried out on the supracrustal successions in the continental rifts related to the final breakup of the Columbia, which has hampered further understanding of the role of the NCC in the Columbia supercontinent. In this contribution, we present integrated detrital zircon U–Pb and Hf isotopic data along with, detrital rutile and whole-rock geochemical studies on the low-grade sedimentary rocks from the Huade Group in the eastern segment of the Zhaertai–Bayan Obo–Huade rift zone in the northern NCC. Combined with other sedimentary and lithostratigraphic data, these new results place significant constraints on the depositional age and provenance of the Huade Group, helping to provide insight into the breakup of the Columbia supercontinent in the northern NCC.

2. Geological background

As one of the oldest cratonic blocks in the world, the NCC was produced by the collision of the Eastern and Western Blocks along the Trans-North China Orogen at ~1.85 Ga (Guo et al., 2005; Zhao et al., 2005, 2012; Kröner et al., 2006; Liu et al., 2006; Wang et al., 2010; Zhang et al., 2012c). After final cratonization, the NCC underwent extensive rifting and anorogenic magmatism in the northern and central parts from 1.78 to 1.68 Ga (Halls et al., 2000; Wang et al., 2004; Yang et al., 2005; Peng et al., 2005, 2007, 2008; Zhang et al., 2007). In the late Paleoproterozoic to Neoproterozoic, a thick sequence of sedimentary rocks was unconformably deposited above the Archean-Paleoproterozoic basement rocks in the Yan-Liao and Zhaertai–Bayan Obo–Huade rift zones in the northern NCC (Fig. 1). Generally, the rift zone was thought to have developed in response to the breakup of the Columbia supercontinent post 1.8–1.6 Ga and the sedimentary rocks were deposited in passive continental margins (He et al., 2000; Huang et al., 2001; Zhu et al., 2005; Hou et al., 2006, 2008; Lu et al., 2008; Kusky and Santosh, 2009; Meng et al., 2011). Recently, zircon ages of K-rich volcanic rocks range from 1.68 to 1.62 Ga have been obtained in the Tuan-shanzi and Dahongyu Formations of the Changcheng Group (Lu and Li, 1991; Li et al., 1995; Lu et al., 2008; Gao et al., 2008a). In the Gaoyuzhuang Formation of the Jixian Group, zircon U–Pb dating on a tuff bed gave an age of 1559 ± 12 Ma (Li et al., 2010). Most recently, the bentonite beds in the Tieling Formation of the Jixian Group and the Xiamaling Formation of the Qingbai Kou Group yielded zircon U–Pb ages of 1437 ± 21 Ma and 1368–1366 Ma, respectively (Gao et al., 2007, 2008b; Su et al., 2008, 2010).

Besides the late Paleoproterozoic to middle Mesoproterozoic sedimentary rocks in the Yan-Liao rift zone, many diabase sills and granitoids previously regarded as Paleozoic in age have been suggested to中间Mesoproterozoic. For example, a diabase sill

Fig. 1. Tectonic subdivision of the North China Craton (modified after Zhao et al., 2005). Also shown is location of the Zhaertai–Bayan Obo–Huade rift zone.

Fig. 2
intruding into the Jixian and Qingbaikou Groups gave zircon and baddeleyite U–Pb ages of 1.35–1.32 Ga (Zhang et al., 2009, 2012a; Li et al., 2009). In the Zhaertai–Bayan Obo–Huade rift zone, the existence of middle Mesoproterozoic granitoids has been proved by the 1.33–1.31 Ga A-type granites in the Shangdu–Huade region (Zhang et al., 2012a; Shi et al., 2012).

Compared with the detailed investigations in the Yan-Liao rift zone, the Zhaertai–Bayan Obo–Huade rift zone has been less studied, possibly due to its strong deformation and poor exposure of the Paleo- to Mesoproterozoic rocks (Wang et al., 1992a). Supracrustal successions in the rift zone were distributed in an E–W trending basin and have been subdivided and given different stratigraphic names in different areas (Qiao, 1991; Wang et al., 1992a). Nevertheless, the Zhaertai, Bayan Obo and Huade Groups are thought to have been deposited roughly simultaneously in the late Paleoproterozoic to early Mesoproterozoic (Wang et al., 1992a). Based on the detrital zircon U–Pb and Hf isotopic data and whole-rock Nd isotopic data from the Zhaertai Group, Li et al. (2007) suggested that the group was derived from the late Archean basement rocks underlying the group and was deposited from ~1.75 Ga during the breakup of the Columbia supercontinent.

The Huade Group is located at the eastern segment of the rift zone and bounded by the Yan-Liao rift zone to the east, the western part of the Hongqiyingzi “Group” and the Khondalite Belt to the south, the Guyang and Wuchuan complexes to the southwest, and the Bayan Obo Group to the west (Fig. 1). The Hongqiyingzi “Group” is a complex of granitic gneisses, migmatites, paragneisses, amphibolites, marbles, quartzites and schists (Li et al., 2007). SIMS and LA-ICP-MS zircon dating results revealed that the “group” has experienced at least four episodes of tectono-thermal events at ~2.50, ~2.18, ~1.85 and 0.4–0.3 Ga (Li et al., 2007). The major lithologies of the Khondalite Belt are khondalite series, TTG gneisses with minor mafic granulites, syntectonic charnockites and S-type granites (Zhao et al., 2005). Recent LA-ICP-MS and SIMS U–Pb zircon data suggest that the protoliths of the khondalite rock series were deposited and metamorphosed in the Paleo-proterozoic, with detrital zircon ages ranging between 2.3 and 1.8 Ga and metamorphic zircon ages at 2.0–1.9 Ga (Wan et al., 2006; Xia et al., 2006a, 2006b; Santosh et al., 2007; Yin et al., 2009, 2011). Other dating techniques have also given similar or younger metamorphic ages, including U–Pb rutile age of 1793 Ma (Wu et al., 1998) and EPMA U–Th–Pb monazite age of 1917 ± 48 Ma (Santosh et al., 2007). On the other way, the Guyang and Wuchuan complexes have traditionally been regarded as a Neoarchean granite-greenstone belt and a Neoarchean granulite-facies complex, respectively (Li et al., 1987). Recent SIMS and LA-ICP-MS U–Pb zircon dating results indicate that granitic and volcanic rocks in both of the two complexes were formed during late Archean and metamorphosed at ~2.5 Ga (Jian et al., 2005, 2012; Chen, 2007; Dong et al., 2012).

Because of the extensive Phanerozoic cover and emplacement of Paleozoic granitoids, the relatively continuous stratigraphic sequences of the Huade Group are only exposed in the northwest of the Kangbao County, the south of the Huade County and the northwest of the Shangdu County (Fig. 2). Generally, the Huade Group was subdivided into the Maohuqing, Gejiaying, Sanxiantian and Huijertu Formations, all of which have been metamorphosed to sub-greenschist facies (Li et al., 2005; Hu et al., 2009). The lowest Maohuqing Formation is >3200 m thick and its main lithologic sequences change from the lower feldspar quartzites and quartz schists, through the middle sericite schists and quartzites, to the upper phyllites (Fig. 3). Conformably overlying the Maohuqing Formation is the Gejiaying Formation that is composed of ~2000 m thick bed of basal pebbled quartzites, quartz schists, quartzites and slates in the lower and ~2300 m thick phyllites, slates, quartz schists and marbles in the middle and upper sequences, interpreted as upward-deepening sequences changing from a coastal to a shallow-marine depositional environment (Fig. 3; Li et al., 2005). The conformably overlying Sanxiantian Formation consists of quartzites, quartz schists and phyllites interlayered with slates in the middle sequence. Widespread cross-bedding and ripple marks preserved in the quartzites suggest a coastal depositional environment (Li et al., 2005). The Sanxiantian Formation is overlain by the Huijertu Formation, which consists mainly of quartzites, marbles, diopsidites and schists (Fig. 3). The overall sedimentary facies of the Huade Group suggest major changes in the basin configuration related to a rifting event in the northern NCC (Li et al., 2005; Hu et al., 2009).

3. Samples and analytical methods

Twenty metasedimentary samples (18 meta-sandstones and two meta-siltstones) were collected from different stratigraphic
positions of the Huade Group for major and trace element analysis. Whole-rock geochemical analyses were performed at the National Research Center for Geoanalysis, Beijing. Major elements were determined by X-ray fluorescence techniques on fused glass beads using a Rigaku-2100. The samples for trace element analysis were prepared in closed beakers in high-pressure bombs to ensure complete digestion and trace element data were acquired by a TJA-PQ-Excel ICP-MS.

Three meta-sandstone samples were also chosen for rutile geochemical analysis by LA-ICP-MS using an Agilent 7500a mass spectrometer equipped with a New-Wave UP-193 at the Geological Lab Center, China University of Geosciences, Beijing. Rutile grains were picked from heavy-mineral residues, after crushing, sieving and standard magnetic and heavy liquid separation, and finally mounted on double sided adhesive tape, enclosed in epoxy resin and polished to about half their size. For LA-ICP-MS analysis, samples were ablated with a spot size of 36 μm at a pulse rate of 10 Hz for 45 s duration. Element concentrations were calculated by the software “GLITTER” using measurements of the following isotopes: 29Si, 48Ti, 53Cr, 90Zr, 95Nb, 178Hf, 181Ta, 208Po, 232Th, 238U, dwell-times were 6–30 ms. 48Ti was used as the internal standard and the rutile was assumed to be stoichiometric with a calculated Ti concentration of 599 343 ppm based on the atomic masses of Ti and O. NIST 610 glass was used as the external standard and analyzed after every 10 sample analyses, with recommended values taken from Pearce et al. (1997). Two formulae of Zr-in-rutile thermometers from Zack et al. (2004) and Watson et al. (2006) (abbreviated as \(T_z\) and \(T_w\), respectively) were applied in this study to calculate the formation temperature of each rutile grain.

Four meta-sandstones and two meta-siltstones were chosen for detrital zircon U–Pb and Hf isotopic analyses. Zircons were separated by standard heavy liquid and magnetic separation methods. Then they were mounted in epoxy resin discs, polished and imaged in both reflected and transmitted light. In order to reveal internal structures of the zircons, CL imaging was carried out by a JSM6510 SEM with attached Gatan CL detector. U–Pb isotopic analyses were performed using a New-Wave UP-213 laser-ablation system coupled with a Thermo-Finnigan Neptune MC-ICP-MS at MRL Key Laboratory of Metallogeny and Mineral Assessment, Institute of Mineral Resources, Chinese Academy of Geological Sciences, Beijing. The 213 nm Arf excimer laser, homogenized by a set of beam delivery groups, was focused on the zircon surface with the energy density of 2.5 J/cm². The ablation protocol employed a spot diameter of 25 μm at 10 Hz repetition rate for 30 s. Helium was used as

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**Fig. 3.** Stratigraphic subdivision of the Huade Group.

Modified after Li et al. (2005).

<table>
<thead>
<tr>
<th>Formation</th>
<th>Lithostratigraphy</th>
<th>Sample</th>
<th>Depositional setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hujiertu</td>
<td></td>
<td></td>
<td>coastal-shallow marine</td>
</tr>
<tr>
<td>Sanxian</td>
<td></td>
<td></td>
<td>coastal</td>
</tr>
<tr>
<td>Gejiaying</td>
<td></td>
<td></td>
<td>coastal-shallow marine</td>
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<tr>
<td>Maohuing</td>
<td></td>
<td></td>
<td>delta-coastal</td>
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![Diagram](image-url)
carrier gas to efficiently transport aerosol to the MC-ICP-MS. Zircon
CJ-1 was used as the external standard while U–Pb raw data were
corrected offline using ICPSMDataCal 8.3 (Liu et al., 2010). More
detailed instrumental setting and analytical procedures have been
described by Hou et al. (2009). Concordia diagrams and binned frequency
histograms were generated using the Isoplot 3.23 program
(Ludwig, 2003) and the results were reported with 1σ errors. In the
data interpretation, zircon ages with 70–105% concordance were
thought to be reliable.

Zircon Hf analyses were also finished at MRL Key Labora-
tory of Metallogeny and Mineral Assessment, Institute of Mineral
Resources, Chinese Academy of Geological Sciences, Beijing, using
the method of Hou et al. (2007). Analyses were measured in situ
with the laser-ablation system New-Wave UP-213 coupled with a
Thermo-Finnigan Neptune MC-ICP-MS. All analyses used a beam
diameter of 55 μm and energy density of 15–201/cm2. Pleso-
vice standard zircon, with a recommended 176Hf/177Hf ratio of
0.282482 ± 0.000013, was used as the reference standard (Slama
et al., 2008). All the Hf isotope analysis results were reported with
an error of 2σ of the mean and values of αHf were calculated using
the decay constant for 176Lu of 1.865 × 10−11 year−1 (Scherer et al.,
2001). Depleted mantle model ages (TDM) were calculated based on
the measured 176Lu/177Hf ratios, referred to the depleted mantle
with present-day 176Hf/177Hf = 0.28325 and 176Lu/177Hf = 0.0384
(Griffith et al., 2000). Crustal model ages (Tc), assuming a mean
crystal value of 176Lu/177Hf = 0.015, were calculated for the source
rock of the magma using the initial 176Hf/177Hf ratio of the zircon
(Griffith et al., 2004).

4. Analytical results

4.1. Whole-rock geochemistry

As mentioned above, most of the metasedimentary rocks in the
Huaode Group have experienced subgreenschist-facies meta-
morphism (Li et al., 2005; Hu et al., 2009), and it is very possible
that whole-rock geochemical compositions, especially major ele-
ments, have been affected by solid state diffusion or mobilizing
elements in a fluid phase (Taylor et al., 1986; Tran et al., 2003).
Furthermore, the complex processes involved in chemical weathering,
mechanical breakdown, and continued chemical alteration during
transport, burial, and diagenesis may also change the whole-rock
geochemistry (McLennan et al., 1993). However, a large number
of geochemical studies have revealed that high field strength ele-
ments (HFSEs) and rare earth elements (REEs) are reliable to be used
as provenance indicators for low-grade metasedimentary rocks
(Winchester and Floyd, 1977; Taylor et al., 1986; Tran et al., 2003;
Wang and Zhou, 2012). Therefore, in this study, HFSEs and REEs
rather than major and other trace elements will be used for inter-
pretation to minimize possible chemical alteration.

Geochemical compositions of the analyzed metasedimentary
samples from the Huaode Group are listed in Supplementary Table
1. The meta-siltstone samples as a whole are relatively immature,
which is reflected by relatively low SiO2 contents (61.3% and 55.6%),
and high Al2O3 contents (10.3% and 16.0%), suggesting petrologic
compositions (Supplementary Table 1). On the sediment classification
diagram of Herron (1988), the meta-siltstone samples were plot-
ted within the shale and arkose fields, respectively (Fig. 4). On the
other hand, the meta-sandstone samples have higher and vari-
able SiO2 contents (61.2–89.8%) and relatively low Al2O3 contents
(2.6–20.2%), protoliths of which range from shale, wacke, arkose,
Fe-sand, sublitharenite, to subarkose, possibly reflecting potassium
metasomatism (Fig. 4; Condie et al., 1992; Pedo et al., 1995).

Supplementary table related to this article can be found, in the online version, at http://dx.doi.org/10.1016/j.precamres.2014.09.011.

Chondrite-normalized REE patterns are shown in Fig. 5 and most of them are characterized by light REEs enrichment rela-
tive to heavy REEs (La/YbN = 5.5–19.8) and negative Eu anomalies
(Eu/Eu* values = 0.48–0.74), similar to that of the upper continen-
tal crust (Taylor and McLennan, 1985). Furthermore, the lack of 
chondrite-normalized negative Ce anomalies rules out significant
hydrothermal alteration (Plank and Langmuir, 1998). It is also wo-
orthy to note that the La/YbN ratios of samples 11HD16–1, 11HD16–2
and 11HD35–1 are 2.9, 3.3 and 1.7, respectively, suggesting higher
inputs of mafic sources than in other samples (Bhatia, 1985).

For trace elements, the Huaode samples are characterized by vari-
able contents of Th (1.5–20.8 ppm), Sc (1.9–25.2 ppm), Zr
(31–414 ppm) and Co (0.1–38.6 ppm). Compared with the post-
Archean Australian shale (PAAS; Taylor and McLennan, 1985),
the metasedimentary samples show little negative anomalies in CaO,
Na2O and Sr, likely reflecting minimal dissolution of plagioclase
(Fig. 6; Mader and Neubauer, 2004). In addition, most of the Huaode
samples have high La/Sc, Th/Sc and La/Co ratios with the exception

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**Fig. 4.** Classification of samples of the Huaode Group after Herron (1988) using log(Fe3O4/K2O) vs. log(SiO2/Al2O3).
of Samples 11HD06-1, 11HD16-1 and 11HD16-2, suggesting more mafic sources than other samples (Supplementary Table 1; Cullers, 2000).

4.2. Rutile geochemistry

The analyzed rutiles from the Huade Group vary in size from 40 to 300 μm and are yellowish to reddish-brown in color. The results of 178 single LA-ICP-MS spot measurements are summarized in Supplementary Table 2.

Supplementary table related to this article can be found, in the online version, at http://dx.doi.org/10.1016/j.precamres.2014.09.011.

Rutiles in the samples 11HD16-1 and 11HD16-2 from the Sanxianian Formations have relatively narrow range of geochemical compositions of Cr (103–595 ppm), Nb (1419–5656 ppm) and Zr (291–3293 ppm), whereas rutiles from Sample 11HD31-1 of the Hujiertu Formation display relatively wide range of geochemical compositions of Cr (629–5353 ppm), Nb (607–4143 ppm) and Zr (482–17995 ppm). In the Nb vs. Cr diagram (Meinhold et al., 2008), rutile geochemical data indicated a mixed source for the Hujiertu Formation and single metapelitic sources for the Sanxianian Formation (Fig. 7a–c).

Results of Zr-in-rutile thermometry are showed as binned histograms in Fig. 7d–f. It is worthy to note that application of Zr-in-rutile thermometry to detrital zircons is based on the assumption of excess ZrO2 and SiO2 in meta-pelites (Zack et al., 2004). In this...
study, calculated temperatures are between ca. 715 and 1040 °C ($T_2$) and ca. 640–890 °C ($T_W$), respectively (Fig. 7d–f). In general, $T_2$ gives a wider range and higher formation temperatures than $T_W$, except for grains with <74 ppm Zr contents (Watson et al., 2006). However, both of $T_2$ and $T_W$ show a change toward “hotter” rutiles from the Sanxianqiao to the Hujiertu Formation (Fig. 7d–f). Furthermore, very fine zircon inclusions in rutile may cause elevated Zr contents and calculated temperatures (Zack et al., 2004). High Zr concentrations in our rutile are not accompanied by high Si contents, we therefore interpret the high temperatures calculated for these grains as real formation temperatures and not due to zircon inclusions.

4.3. U–Pb zircon ages

U–Pb zircon ages of five meta-sandstones and one meta-siltstone collected from the four formations of the Huade Group were studied. All analytical data are presented in Supplementary Table 3 and detrital zircon ages with concordance of 70–105% are thought to be reliable and accepted for binned frequency histograms and $\varepsilon_{Hf}$ vs. $^{207}$Pb/$^{206}$Pb age diagrams.

4.3.1. Maohuqing Formation

Sample 11HD10-1 is quartzite collected from the upper succession of the Maohuqing Formation (Fig. 3). Zircons have relatively small sizes (50–100 μm) and subrounded to well-rounded shapes (Fig. 8a–d). A total of 80 analyses yielded 75 usable ages (Supplementary Table 3). With the exception of one older grain with the age of 2865 Ma, nearly all the grains lie within the age range between 2639 and 1855 Ma. Within the major age range, there is a major age peak at ~1895 Ma and a subordinate age peak at ~2520 Ma, which are defined by 29 and 23 zircon ages, respectively (Fig. 9a).

Supplementary table related to this article can be found, in the online version, at http://dx.doi.org/10.1016/j.precamres.2014.09.011.

4.3.2. Gejiaying Formation

Sample 11HD01-2 is quartzite collected from the lower succession of the Gejiaying Formation (Fig. 3). Zircons are variable in size and shape ranging from small (80 μm) round to large (200 μm) prismatic grains (Fig. 8e–h). From a total of 80 analyses, two discordant analyses are excluded for the further discussion (Supplementary Table 3). Of the remaining 78 zircon ages, except one grain with an older age of 2651 Ma, all others yielded concordant ages ranging from 2587 to 1857 Ma with a major peak at ~1960 Ma and a minor peak at ~2550 Ma, which are defined by 45 and 14 zircon ages, respectively (Fig. 9b).

Sample 11HD07-1 is fined-grained quartzite from the middle succession of the Gejiaying Formation (Fig. 3). Zircons range in length from 100 to 200 μm and most of them show banded or oscillatory zoning structures (Fig. 8i–l). A total of 80 analyses were performed and all of them gave reliable ages (Supplementary Table 3). The binned frequency histogram of these analyses shows that most grains lie within the age range of 2698 and 1847 Ma with the exception of two older grain with ages of 2740 and 2855 Ma. Within the age range, there is one major age peak at ~1880 Ma and a minor
age peak at ~2540 Ma, which include 42 and 11 analytical results, respectively (Fig. 9c).

4.3.3. Sanxian Formation

Sample 11HD06-1, a fine-grained sericite quartzite, was collected from the lower succession of the Sanxian Formation. Generally, smaller zircons (~30 µm) are round and larger zircons (~100 µm) are elongate or prismatic in this sample (Fig. 8m–p). From a total of 80 analyses, one discordant analysis was excluded (Supplementary Table 3). Of the remaining 79 zircon grains, except four older grains with ages of 3102–2714 Ma and five younger grains with age of 1444–1328 Ma, most grains lie within the age range between 2618 and 1510 Ma. Inspection of the histogram shows there is a major peak at ~1890 Ma (18 grains), with one shoulder at ~2530 Ma (16 grains) and one minor peak at ~1780 Ma (13 grains; Fig. 9d). The youngest age peak formed by two grains yielded the weighted average age of 1333 ± 17 Ma (MSWD = 0.74).

Sample 11HD16-1 is a fine-grained quartz schist collected from the middle succession of the Sanxian Formation (Fig. 3). Zircons are not plentiful in the heavy mineral fraction and have relatively small size and round shape (<80 µm; Fig. 8q–t), implying long-distance transport. A total of 80 analyses yielded 70 usable ages (Supplementary Table 3). The age histogram of this sample is different from other samples from the Huade Group, with most zircon

Fig. 8. Representative selection of cathodoluminescence (CL) zircon images. Circles (55 and 25 µm) show positions of Hf and U–Pb analytical sites. $^{207}$Pb/$^{206}$Pb and $\epsilon_{\text{Hf}}(t)$ values are also plotted. The scale bar is 100 µm long.
grains showing ages between 1643 and 1451 Ma and a major age peak of ~1500 Ma defined by 58 analytical results (Fig. 9e). The youngest age peak formed by three grains yielded the weighted average age of 1368 ± 31 Ma (MSWD = 0.069).

4.3.4. Huijerti Formation

Sample 11HD31-1 is a sericite quartz schist from the lower succession of the Huijerti Formation (Fig. 3). A total of 80 spot analyses were measured in this sample and nearly all of them are concordant or nearly concordant (Supplementary Table 3). Fig. 9f shows the binned frequency histogram for 78 of all the analyses, all between 70% and 105% concordant. With the exception of eight older grains with ages between 2573 and 2051 Ma, most grains scatter within the age range of 1917 and 1222 Ma. Within the age range, the major age peak of ~1490 Ma is defined by 16 zircon ages (Fig. 9f). The youngest zircon identified in this sample is grain 15 that yields an age of 1222 ± 13 Ma with concordance of 92% (Supplementary Table 3), whereas the youngest age peak formed by nine zircons gives the weighted average age of 1337 ± 7 Ma (MSWD = 0.22).

4.4. Zircon Hf isotopes

From five meta-sandstone samples (11HD01-2, 11HD06-1, 11HD07-1, 11HD16-1, 11HD31-1) and one meta-siltstone sample (11HD10-1), zircons with 70–105% concordance U–Pb ages were chosen for Hf isotopic analyses. The analyzed zircons with 176Yb/177Hf ratios more than 0.15 and display positive linear relationship between 176Yb/177Hf and 176Hf/177Hf ratios are excluded for further calculation and discussion and other results are listed in Supplementary Table 4 and presented in Fig. 10 on the ε_Hf vs. 207Pb/206Pb age plots.

Supplementary table related to this article can be found, in the online version, at http://dx.doi.org/10.1016/j.precamres.2014.09.011.

An older age population (2690–2458 Ma) defined by zircons in Samples 11HD01-2, 11HD07-1 and 11HD10-1 from the lower part of the Huahe Group, predominantly exhibit a range of ε_Hf values from −3.9 to +8.7 (Fig. 10a–c) and T_c model ages of 2.45–3.37 Ga, suggesting reworking of an older crust and juvenile crustal additions. A younger population (2171–1770 Ma) of these three samples...
plots within a relatively wide range of $\varepsilon_{\text{Hf}}$ values from $-7.8$ to $+8.3$, with $T_R$ model ages of 2.14–3.22 Ga, indicative of a mixing of the older crust with juvenile materials (Fig. 10a–c). Zircons from Sample 11HD06-1 display two age populations (2650–2458 and 2158–1750 Ma) similar to samples from the lower part of the Huade Group, with nearly the same range of $\varepsilon_{\text{Hf}}$ values and $T_R$ model ages (Fig. 10d). Furthermore, for the younger population (1700–1305 Ma) in Sample 11HD06-1, most zircons possess positive $\varepsilon_{\text{Hf}}$ values, suggesting dominant inputs of juvenile crust with limited reworking of an older crust (Fig. 10d). In Sample 11HD31-1, zircons with ages between 1932 and 1707 Ma exhibit similar $\varepsilon_{\text{Hf}}$ values to those of similarly ages zircons from the Maohuqing and Gejiaying Formations, whereas younger zircons of 1627–1297 Ma display similar $\varepsilon_{\text{Hf}}$ values to the youngest age population of Samples 11HD06-1 (Fig. 10f).

5. Interpretations and discussion

5.1. Provenance of the Huade Group

Geochemically, most of the Huade sedimentary rocks have REE patterns similar to that of the upper continental crust, showing light REEs enrichment relative to heavy REEs and negative Eu anomalies (Fig. 5; Taylor and McLennan, 1985). In addition, high La/Sc, Th/Sc and La/Co ratios in these samples indicate that the major provenance of the Huade Group was composed of felsic to intermediate rocks (Cullers, 2000). However, two samples (11HD16-1 and 11HD16-2) from the Sanxian Formation have depleted light REEs and slightly enriched heavy REEs compared to the upper continental crust and low La/Sc, Th/Sc and La/Co ratios, suggesting significant inputs of mafic components (Cullers, 2000).

U–Pb ages of detrital zircons from the metasedimentary rocks of the Huade Group place more constraints on their source rocks. From a K–S test (Press et al., 1988) for detrital zircon ages of samples in this study and data from Hu et al. (2009), $P$-values are higher than 0.08 for sample pairs of 11HD06-1/06KB18, 06SD12/06SD01, 11HD01-2/11HD07-1 and 11HD10-1/11HD01-2 because of the similar age peaks at $\sim 1.9$ and $\sim 2.5$ Ga, most likely suggesting statistically indistinguishable provenance. By contrast, when comparing the detrital zircon ages of Samples 11HD16-1 and 11HD31-1 with the other samples, the calculated $P$ values are near to 0. In Fig. 9e and f, one can see that the dominance of the $\sim 1.5$ Ga detrital zircons of the two samples from the upper two formations is the main reason leading to the low values of $P$. Therefore, U–Pb ages of all detrital zircons in this study are combined with data from Hu et al. (2009) based on the formation and displayed in binned frequency histograms (Fig. 11), which provide more information about the source rocks.

Detrital zircons from the metasedimentary rocks of the Huade Group exhibit a major age range between 2690 and 2450 Ma, with a peak at $\sim 2520$ Ma (Fig. 11). This age range is well consistent with the ages of the diorites and granitoids (2556–2520 Ma; Jian et al., 2005, 2012) and the metadacites in the Guyang complex (2516–2500 Ma; Chen, 2007), the orthopyroxene-bearing TTG gneisses in the Wuchuan complex (2545–2507 Ma; Dong et al., 2012) and the orthogneisses of the Hongqiyingzi “Group” (2535–2484 Ma; Liu et al., 2007) from the Yinshan Block. Available geochemical and geochronological data indicate that the episodic
A subordinate population of detrital zircons from the Huade Group has an age range between 2150 and 1710 Ma, with a peak at ~1900 Ma (Fig. 11). The intermediate to felsic rocks with such an age range, although volumetrically subordinate, are reported in the adjacent Hongyijingzi “Group”, represented by the granitic mylonite gneisses, amphibole-biotite-quartz dioritic gneisses and biotite-monzonitic granitic gneisses (1871–1810 Ma; Liu et al., 2007). In addition, in the adjacent Khondalite Belt, a large number of similar aged Paleoproterozoic detrital zircons have been reported, which have an age range from 2150 to 1830 Ma, with the peak at ~1950 Ma (Wan et al., 2006; Xia et al., 2006a, 2006b; Yin et al., 2011). These detrital zircons from the Khondalite Belt yielded εHf values ranging from −7.9 to +9.7, which overlap with the εHf values of these Paleoproterozoic detrital zircons from the Huade metasedimentary samples (Fig. 10), suggesting that the subordinate part of the group was derived from the erosion of the Paleoproterozoic granites from the Hongyijingzi “Group” and/or recycled from the metasedimentary units from the Khondalite Belt.

On the other hand, relatively subordinate zircons with the ages of 1660–1330 Ma (the peak at ~1500 Ma; Figs. 9 and 11) from the Sanxiatian and Huijietu Formations of the Huade Group most likely have been transported from exotic sources. As mentioned above, in the Yan-Liao rift zone to the east, zircon ages of 1.68–1.62 Ga (Lu and Li, 1991; Li et al., 1995; Lu et al., 2008; Gao et al., 2008a), ~1.56 Ga (Li et al., 2010), ~1.44 Ga and ~1.37 Ga of volcanic interlayers (Gao et al., 2007, 2008b; Su et al., 2008, 2010) and 1.35–1.32 Ga diabase sills (Zhang et al., 2009, 2012a; Li et al., 2009) have been reported in the Changcheng, Jixian and Qingshui Group. Moreover, these three groups are composed of dominantly metasedimentary rocks and detrital zircons from them are characterized by a bimodal age peaks of ~2.5 and ~1.8 Ga (Wan et al., 2003, 2011). Therefore, the thickness and zircon fertility of the volcanic interlayers and diabase sills are both much less than those of the metasedimentary rocks (Gao et al., 2008b; Zhang et al., 2009; Su et al., 2010). Therefore, detrital zircons sourced from the Yan-Liao rift zone should be characterized by two major age peaks of ~2.5 and ~1.8 Ga and minor populations of 1.68–1.32 Ga, but this is not the case for the upper part of the Huade Group (Figs. 9 and 11). Therefore, felsic rocks from cratonic blocks connected to the northern margin of the NCC in the Columbia supercontinent are candidate sources for these early Mesoproterozoic detrital zircons from the upper Huade Group. As already mentioned, the suggested adjacent craton to the NCC ranges from the India (Zhao et al., 2002, 2004), through the Baltica (Rogers and Santosh, 2009), to the North America (Hou et al., 2008). In the Indian Craton, the 1480–1262 Ma alkaline complexes have only been reported from the suture zone between the Bhandara craton and the Eastern Ghats Belt and no lithologies or zircons with the age of ~1.5 Ga have been found (Upadhyay, 2008 and references therein). Furthermore, detrital zircons from both the Eastern Ghats Belt and the Chhattisgarh Basin within the Bastar Craton lack the age peak of ~1.5 Ga (Upadhyay et al., 2009; Bickford et al., 2011), suggesting scarce supply of early Mesoproterozoic detritus from the Indian Craton. Therefore, detrital zircons within the age range in the upper Huade Group were not likely derived from the Indian Craton. On the other hand, the 1.5–1.4 Ga Telemark Group in the NW Baltic craton has a detrital zircon age peak at ~1500 Ma, with εHf values from −0.5 to +7.2 (Lamminen and Köykkä, 2010) and detrital zircons from the Mississippi river in the North American craton are characterized by a 1.6–1.3 Ga age peak with εHf values from 0 to +5 (Iizuka et al., 2010). In addition, 1.65–1.50 Ga convergent margin-related magmatism was widespread along the southern and northwestern margins of the North American and Baltic cratons, respectively (Rivers, 1997; Bogdanova et al., 2008). Most early Mesoproterozoic detrital zircons from the studied samples have εHf values ranging between −1 and +10, which are broadly consistent with those of the similar-aged detrital zircons found in the North
American and Baltica cratons, suggesting that the minor part of the upper Huade Group was likely derived from the above two cratonic blocks connecting to the northern margin of the NCC in the Columbia supercontinent.

The chemistry and thermometry of detrital rutiles from metasedimentary rocks of the Huade Group have also provided important information about their source rocks. Rutile Cr–Nb systematic indicated a single input of metapelitic lithologies in the Sanxianxian Formation and a mixed metamafic and metapelitic lithologies in the Huijiertu Formation (Fig. 7). The calculated rutile formation temperatures ($T_{rH}$) range between ca. 640 and 740 °C with a single 890 °C rutile in the Sanxianxian Formation. For the rutiles from the Huijiertu Formation, they gave temperatures between 680 and 760 °C with two hotter grains at ∼900 °C. Based on the detrital zircon age and HF isotopic signatures, we suggest the North American and/or Baltica cratons as their possible sources, so the amphibolite- and eclogite-facies rocks in the Trans-Hudson Orogen (Hoffman, 1989), the Kola-Karelia Orogen (Bogdanova, 1999) or their age-equivalent orogens may be their sources. Another potential source could be the Khondalite Belt, where amphibolite- and eclogite-facies rocks have been reported (Zhao et al., 2005) and metamorphic rutiles from ultra-high-temperature granulites gave similar results (Jiao et al., 2011).

5.2. Depositional age of the Huade Group

Depositional age of the Huade Group has not been well constrained because of the lack of volcanogenic interlayer and radioactive diagenetic minerals. Based on the appearance of small shelly fossils in the upper part of the group, some researchers have suggested that the Huade Group was formed in early Paleozoic (Wang et al., 1992b; Chen, 1993). However, authenticity and geological significance of the fossils have been doubted or even denied by following studies (Li and Zhang, 1993; Zhang, 1994). Recently, Zheng et al. (2004) and Li et al. (2005) suggested that the Huade Group was deposited in the Paleoproterozoic based on their interpretation that the 2.1–1.5 Ga monzogranites intrude the group. However, due to the unclear contact relationship of the monzogranites with the group, their suggestions on the depositional ages of the Huade Group may be questionable.

Our U–Pb dating results of detrital zircons from different lithotragriugraphic units of the Huade Group place reliable constraints on the maximum depositional ages of the group. In this study, 459 detrital zircons from the four formations of the Huade Group were dated. The low greenschist-facies metamorphism of the group, relatively high Th/U ratios (0.1–3.5) and oscillatory zoning structures (Fig. 8) of the analyzed zircons suggest that their U–Th–Pb systems remained closed after their deposition and the youngest detrital zircons ages could be used to constrain the maximum depositional times.

For the Sanxianxian Formation, the youngest group of detrital zircons from Samples 11HD06-1 and 11HD16-1 yielded the weighted average ages of 1333 ± 17 Ma and 1368 ± 31 Ma, respectively, which are consistent within analytical errors. For the overlying Huijiertu Formation, the youngest group of detrital zircons in Sample 11HD31-1 define a weighted average age of 1337 ± 7 Ma, older than the single-grained youngest age of 1222 ± 13 Ma. We favor the age of ~1337 Ma as a more reliable constraint on the maximum depositional age of the Huijiertu Formation based on the following considerations: (1) the youngest age peak of ~1337 Ma was defined by nine grains, which is in accordance with the criterion that the reliable youngest zircon age must belong to a significant population composed of three or more grains (Andersen, 2005; Gehrels et al., 2006); (2) detrital zircons with the U–Pb age of ~1220 Ma do not occur in other studied samples, whereas ~1337 Ma detrital zircons have also been found in the samples from the Sanxianxian Formation.

On the other hand, the Gejiaying and Sanxianxian Formations are cut by the A-type granites, which were dated at ~1320 Ma (Zhang et al., 2012a; Shi et al., 2012). Taken together, the depositional age of the upper Huade Group can be constrained in the period between ~1.34 and ~1.32 Ga.

For the Maohuqing and Gejiaying Formations, the youngest zircon ages found in the studied samples gave similar ages of ~1850 Ma. It is worthy to note that the youngest zircons from the Jixian and Qinghaiakou Groups in the Yan–Liao rift zone also yielded the similar youngest ages of ~1820 Ma (Wan et al., 2003, 2011), whereas the two groups were deposited after 1.6 Ga (Gao et al., 2007; Su et al., 2008, 2010; Li et al., 2010). Such a phenomenon is consistent with the inferred intraplate and divergent plate tectonic setting for these sedimentary units, which usually lack inputs from contemporaneous igneous sources and show an age gap up to 500 Ma between depositional age and the youngest detrital zircon ages (Cawood et al., 2007). Therefore, a conservative constraint on the maximum depositional age of the lower Huade Group given by the youngest detrital zircons is ~1850 Ma.

5.3. Evolution of the Huade Group

Generally, the Paleo-Mesoproterozoic successions in the Zhaeretai–Bayan Obo–Huade and Yan-Liao rift zones on the northern margin of the NCC were believed to be deposited in continental rift zones related to the breakup of the Columbia supercontinent (Zhai and Liu, 2003; Zhao et al., 2003; Lu et al., 2008). The continental rift interpretation has been supported by the bimodal volcanism (Zhang et al., 2012a) and narrow zones of thick sediment accumulation (Wang et al., 1992a). Depositional patterns in continental rift basin are affected by local sites of uplift and erosion, sediment supply and transport, and accommodation space for deposition (Gawthorpe and Leeder, 2000; Lamminen and Koyckk, 2010). In this section, a model based on the combination of provenance and lithostratigraphic data is presented to describe the evolution of the basin in which the Huade Group was deposited.

Evolution of the basin was initiated with syn-rift sedimentation of the lower Maohuqing Formation, which was characterized by coarse-grained delta sediments. Accelerated subsidence was recorded by the upper Maohuqing and Gejiaying coastal-shallow marine deposits, such as quartz schists and phylmites (Li et al., 2005). Synchronously with rifting, the rift flanks possibly underwent local and transient uplift due to the increased sediment loads (Burov and Cloetingh, 1997), leading to the basal pebbly quartzite deposits in the Gejiaying Formation. Detrital zircon data suggest that sediments of the Maohuqing and Gejiaying Formations were derived from the Neoarchean to Paleoproterozoic granitic rocks in the Yinchuan Block and recycled from the metasedimentary rocks of the Khondalite Belt close to the basin. This is also supported by the whole-rock geochemistry because most samples from the lower Huade Group were plotted in or near to the passive margin area, indicating craton-interior provenance (Fig. 12; Bhatia and Crook, 1986).

Subsequent deposition of the Sanxianxian schists, phylmites and minor slates suggests that the subsidence rate of the rift had decreased, with the comeback of a coastal environment (Li et al., 2005). Thermal subsidence became obvious again lately and resulted in marine transgression and shallow-marine carbonate deposition in the Huijiertu Formation (Fig. 3). In this stage, associated with extension leading to continental breakup and sea opening, drainage was established from other cratonic blocks (the Baltica or/and North American cratons), therefore extrabasinal or long-traveled sediments were allowed to enter the basin. This process is readily apparent from the zircon age pattern from the Sanxianxian and Huijiertu Formations, which are different from that of the underlying Maohuqing and Gejiaying Formations (Fig. 11).
This is also supported by the whole-rock geochemistry because most samples from the upper Huade Group were plotted into the continental island arc or active continental margin areas, indicating more inputs of magmatic arc products (Fig. 12; Bhatia and Crook, 1986).

5.4. Implications for the breakup of the Columbia supercontinent

As mentioned in the introduction, controversies still remain about the role of the NCC in the Columbia supercontinent. Firstly, position of the NCC varies in the different configurations of the Columbia supercontinent. Rogers and Santosh (2009) correlated the North Hebei Orogen with the Volyn belt of Baltica, whereas Hou et al. (2008) suggested the North Hebei Orogen as an Andean-type continental margin similar to the Wopmay Orogen of western Canada. On the other way, Zhao et al. (2002, 2004) correlated the 1.9–1.8 Ga active Trans-North China Orogen with the Central Indian Tectonic Zone. Secondly, it is generally believed that the NCC was not involved in the 1.3–1.2 Ga final fragment of the Columbia supercontinent because middle Mesoproterozoic rift-related tectonic events were scarce (Rogers and Santosh, 2002; Hou et al., 2008).

Determination of depositional ages and provenance of the Huade Group in this study places a rigorous constraint on the timing of the breakup of the Columbia supercontinent and its reconstruction. The existence of 1.35–1.31 Ga bimodal magmatic associations caused by continental rift in the northern NCC (Zhang et al., 2009, 2012a; Shi et al., 2012), combined with the 1.34–1.32 Ga upper Huade Group indicate that the breakup of the NCC from the Columbia supercontinent was at middle Mesoproterozoic. These ages are nearly synchronous with those of rift-related activities in other cratonic blocks such as North America and Baltica (Karlstrom et al., 2001; Bogdanova et al., 2008), Greenland (Pearce and Leng, 1996; Upton et al., 2003) and Australia (Wingate et al., 2005). Furthermore, our data also suggest that the northern margin of the NCC has received sediments from the North America and/or Baltica in the middle Mesoproterozoic, which are also supported by the similar paleomagnetic poles of the NCC with the North America, Siberia and Baltica (Pei et al., 2006; Zhang et al., 2012b) during late Paleoproterozoic to early Mesoproterozoic, suggesting that these cratonic blocks joined together at ~1.8 Ga and did not fragment until ~1.38 Ga.

6. Conclusions

(1) Geochemistry of the lower Huade Group indicates that their source rocks are felsic, displaying passive margin signatures. The dominant 2690–2450 detrital zircons were mostly probably sourced from the granitic rocks in the Yinson Block. The subordinate 2150–1710 Ma detrital zircons were probably derived from the erosion of the Paleoproterozoic granites of the Honqiyinzi “Group” and/or recycled from the metasedimentary units from the Khondalite Belt. The youngest detrital zircon age peak of ~1850 Ma in the lower Huade Group indicates that it was deposited after this time.

(2) The metasedimentary rocks from the upper Huade Group have continental island arc or active continental margin geochemical signatures. Besides the age population of 2690–2450 and 2150–1710 Ma, minor amounts of 1660–1330 Ma detrital zircons in the upper Huade Group were likely derived from the North American and/or Baltica cratons connecting to the northern margin of the NCC in the Columbia supercontinent. The youngest age peak of ~1337 Ma, in combination with the ~1320 Ma granitoids intruding the Huade Group, indicates that the depositional ages of the upper Huade Group can be constrained in the period between ~1.34 and ~1.32 Ga.

(3) Detrital rutiles from the Huade Group are suggestive of dominantly metapelitic rocks and peak metamorphic temperature of 700–750 °C.

(4) Our new geochronological and geochemical data suggest that continental rift basin deposits represented by the upper Huade Group on the northern margin of the NCC developed between ~1.34 and ~1.32 Ga and received detritus from the North America and/or Baltica, which indicate that the final breakup of the NCC from the Columbia supercontinent happened in the middle Mesoproterozoic.

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