



# New Detrital Zircon Age of the Paleo-Mesoproterozoic Xiong'er Group, Southern North China Craton

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## INTRODUCTION

The tectonic setting of the southern margin of the North China Craton (NCC) during Paleo-Mesoproterozoic is still under debate (Zhao et al., 2004; He et al., 2009; Zhao et al., 2009; Zhai et al., 2015). One of the controversies regarding this issue is the duration of the Xiong'er volcanism. The Xiong'er Group is mainly composed of basaltic andesite, andesite, dacite, and rhyolite with intercalated siliciclastic rocks. One suggestion is that the Xiong'er volcanism spans a very short period within 1.78–1.75 Ga, typical of an igneous event in a continental rift setting (Zhao et al., 2004; Zhai et al., 2015). By contrast, other researchers consider the duration of the Xiong'er volcanism lasted from ca. 1.78 Ga until 1.45 Ga and suggest a prolonged subduction in the southern NCC during that time (He et al., 2009; Zhao et al., 2009). Here, we present new geochronological data for the interlayered siliciclastic rocks from the Majiahe Formation in the upper Xiong'er Group. Our results show unexpected young zircons at ca. 1400 Ma, possibly suggesting the long-term duration of the Xiong'er volcanism.

## METHODS AND ANALYTICAL PROCESS

### Sample Preparation

The Paleo-Mesoproterozoic succession in the southern NCC is distributed in three sub-regions, namely, the Song-Ji, Mianchi-Queshan, and Xiaoqinling-Luanchuan areas (**Figure 1**). In the Mianchi-Queshan area, the Xiong'er Group is thought to be the earliest deposition during the breakup of the supercontinent Nuna/Columbia. Our sample site is located in the Mianchi-Queshan area. The sample NOD-139-1 was collected from a layer intercalated in volcanic rocks of the Majiahe Formation in the upper Xiong'er Group (**Figure 1**).

The tuffaceous siltstone, which is mainly composed of clay matrix, ilmenite, quartz, and plagioclase, was sampled for zircon geochronological analysis. The separation of the zircons was accomplished at the Langfang (Yuneng) Yuheng Mineral and Rock Service Ltd. and is consistent with the guidelines described in Liu et al. (2018). The sample was crushed into 40–100 mesh after removing inclusions and veins. Then, rock powder was elutriated and separated into the light and heavy segments, which the latter segments were processed with the strong magnetic selection. The less magnetic portion was then filtered by the electromagnetic filter, in which the non-magnetic minerals will be detained for the heavy liquid filter. The metal sulfide in the products was further removed by the high-frequency dielectric separator, leaving the portion that contains mostly zircon. Last, the zircons were hand-picked and purified by a binocular microscope.

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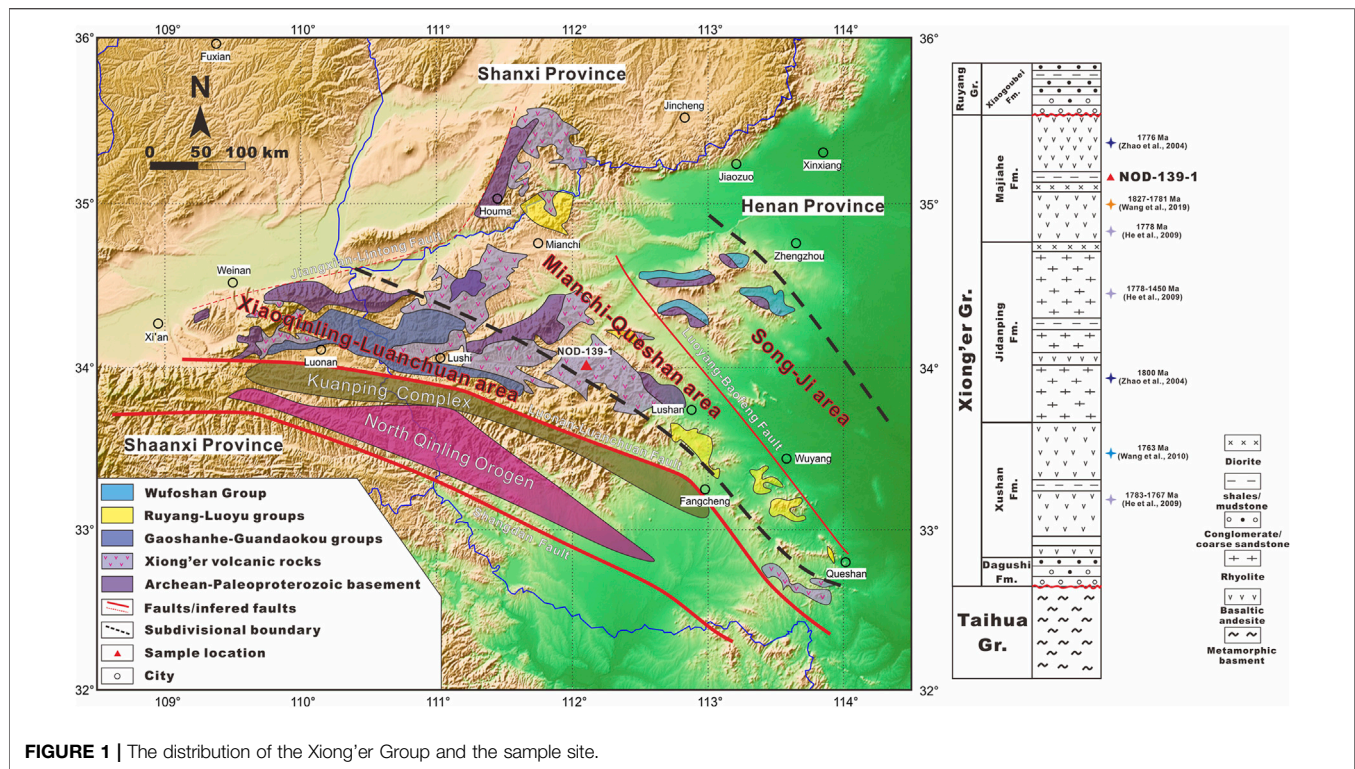
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Two hundred randomly hand-picked zircon grains were mounted in epoxy and polished to approximately half of the average zircon grain thickness. The morphological structure was examined by transmission and reflection light microscopy. The internal structure of zircons was examined using cathodoluminescence (CL) images. Zircon CL images were obtained using an Analytical Scanning Electron Microscope (JSM IT100) connected to a GATAN MINICL system. The imaging condition was 10.0–13.0 kV voltage of electric field and 80–85  $\mu\text{A}$  current of the tungsten filament.

## Analytical Process

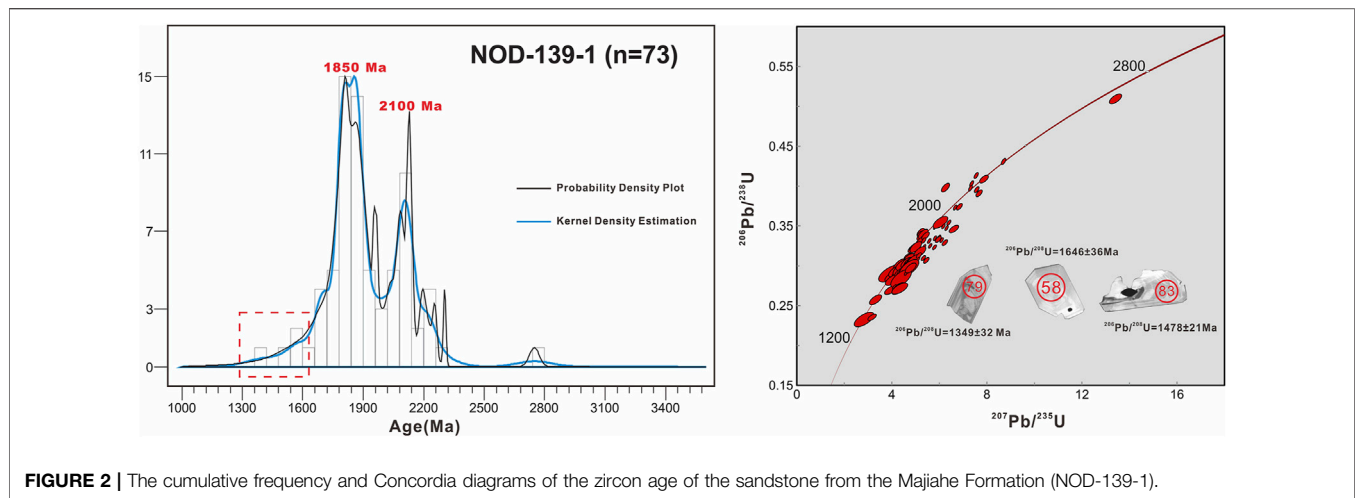
The zircon U-Pb isotope was analyzed by laser-ablation-inductively coupled plasma-mass spectrometry (LA-ICPMS) using an Agilent 7,500a mass spectrometer equipped with GeoLas 200 M laser-ablation system consisting of CompEx102 (193 nm ArF-excimer laser, Lambda Physik) and an optical system (MicroLas) at the National Key Laboratory of Continental Dynamics in the Northwest University, China. The spot size was 25  $\mu\text{m}$ , and the carrier gas helium was transported into the ablation cell. All the experimental procedures are referred to Liu et al. (2007) and Liu et al. (2018). In this study, the U-Pb isotopic ages and the instrumental mass deviation were calibrated by using the standard zircon 91,500 and GJ-1, respectively. The analysis on the standard reference zircon 91,500 was conducted in every six spots while the GJ-1 was in every twelve spots. The weighted mean ages of the 91,500 and GJ-1 during this process were presented in the supplementary materials (Supplementary Figure S1). The widely used 91,500

reference zircon was dated with the isotope-dilution thermal-ionization (ID-TIMS)  $^{206}\text{Pb}/^{238}\text{U}$  age of  $1,065.4 \pm 0.6$  Ma (Wiedenbeck et al., 1995). In the National Key Laboratory of Continental Dynamics in the Northwest University, previous studies yielded the LA-ICP-MS  $^{206}\text{Pb}/^{238}\text{U}$  age of  $1,062.2 \pm 6.0$  Ma (Yuan et al., 2008). In this study, the weighted mean  $^{206}\text{Pb}/^{238}\text{U}$  age of the 91,500 is  $1,062.6 \pm 4.2$  Ma, within the  $2\sigma$  error of the ID-TIMS age. The reference zircon GJ-1 was first analyzed with the ID-TIMS  $^{207}\text{Pb}/^{206}\text{Pb}$  and  $^{206}\text{Pb}/^{238}\text{U}$  ages at  $608.5 \pm 0.4$  Ma and  $599.8 \pm 4.5$  Ma, respectively (Jackson et al., 2004). The previous study in this laboratory yielded a slightly older  $^{206}\text{Pb}/^{238}\text{U}$  age of  $604.6 \pm 2.9$  Ma (Yuan et al., 2008). Our analysis yields an intercept age of  $607 \pm 6.3$  Ma and a weighted mean  $^{206}\text{Pb}/^{238}\text{U}$  age of  $608.6 \pm 6.9$  Ma, respectively (Supplementary Figure S1).

To reduce the probability of missing age components, 84 zircon grains were randomly selected for analysis in this study (Dodson et al., 1988; Fedo et al., 2003). The concentration ratios and the apparent ages were calculated using ICPMSDataCal (Liu et al., 2008; Liu et al., 2010). The common lead calibration was also applied according to Andersen (2002). Detrital zircon age spectra were calculated and presented using ISOPLOT (version 3.23) (Ludwig, 2003) and the DensityPlotter (Vermeesch, 2012).

## CONCLUSION

The 84 spots yield 73 single-grain data that are less than 10% discordant. The external morphology of the zircons is variable



**FIGURE 2 |** The cumulative frequency and Concordia diagrams of the zircon age of the sandstone from the Majiahe Formation (NOD-139-1).

from anhedral with a nearly rounded shape to euhedral with a clear pyramid face while most are subhedral. Some of the grains are the fragments of intact ones (e.g., the No.10 grain in **Supplementary Figure S2**). From the cathodoluminescence intensities, the zircon grains of this sample can be divided into two groups (**Supplementary Figure S2**). One group with dark and strong CL emission showed homogeneous or patchy internal texture whereas another group with weak to intermediate intensities showed distinct oscillatory zones which indicated the igneous origin (**Supplementary Figure S2**). Meanwhile, the former group generally yields older age which corresponds with high U content and lower Th/U ratio (**Supplementary Figure S2**; **Supplementary Table**). These zircons are possibly from the metamorphic basement or the recycled sedimentary rocks. The zircons with more white color and oscillatory zones show relatively younger ages. The three youngest zircon grains showed distinct euhedral external morphology and blurred internal oscillatory zones (**Figure 2**). The probability density distribution of the obtained zircon ages shows two prominent peaks at 2,100 Ma and 1850 Ma (**Figure 2**). The former is possibly sourced from the Taihua (tonalite-trondhjemite-granodiorite) 2000–2,200 Ma TTG assemblages in the southern NCC while the latter is consistent with the ca. 1850 Ma granitic pluton in the central NCC (Huang et al., 2010; Zhao and Zhai, 2013). There are a considerable number of zircon grains that yield ages younger than the previous geochronological reports for the Xiong'er Group, among which the three youngest  $^{206}\text{Pb}/^{238}\text{U}$  ages are  $1,349 \pm 32$  Ma,  $1,478 \pm 21$  Ma, and  $1,646 \pm 36$  Ma (**Figure 2**). Our new detrital zircon age report from the Majiahe Formation in the upper Xiong'er Group provides a new constraint on the duration of the Xiong'er volcanism in the southern NCC. According to previous studies, if the Xiong'er volcanism occurred in a short duration from 1.78 Ga to 1.75 Ga, it is impossible to get younger age from the interval of the Xiong'er Group. Therefore, our data in this study contradicted the opinion above whereas supported the model proposed by

He et al. (2009) and Zhao et al. (2009), in which the Xiong'er volcanism probably can last to 1,450 Ma or even younger.

## DATA AVAILABILITY STATEMENT

The original contributions presented in the study are included in the article/**Supplementary Material**, and further inquiries can be directed to the corresponding author.

## AUTHOR CONTRIBUTIONS

XL acquired and processed the data, wrote, and drawn the pictures.

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## SUPPLEMENTARY MATERIAL

The Supplementary Material for this article can be found online at: <https://www.frontiersin.org/articles/10.3389/feart.2021.732505/full#supplementary-material>

## REFERENCES

- Andersen, T. (2002). Correction of Common Lead in U-Pb Analyses That Do Not Report  $^{204}\text{Pb}$ . *Chem. Geology*. 192, 59–79. doi:10.1016/S0009-2541(02)00195-X
- Dodson, M. H., Compston, W., Williams, I. S., and Wilson, J. F. (1988). A Search for Ancient Detrital Zircons in Zimbabwean Sediments. *J. Geol. Soc.* 145 (6), 977–983. doi:10.1144/gsjgs.145.6.0977
- Fedo, C. M., Sircombe, K. N., and Rainbird, R. H. (2003). Detrital Zircon Analysis of the Sedimentary Record. *Rev. Mineralogy Geochem.* 53 (1), 277–303. doi:10.2113/0530277
- He, Y., Zhao, G., Sun, M., and Xia, X. (2009). SHRIMP and LA-ICP-MS Zircon Geochronology of the Xiong'er Volcanic Rocks: Implications for the Paleo-Mesoproterozoic Evolution of the Southern Margin of the North China Craton. *Precambrian Res.* 168, 213–222. doi:10.1016/j.precamres.2008.09.011
- Huang, X.-L., Niu, Y., Xu, Y.-G., Yang, Q.-J., and Zhong, J.-W. (2010). Geochemistry of TTG and TTG-like Gneisses From Lushan-Taihua Complex in the Southern North China Craton: Implications for Late Archean Crustal Accretion. *Precambrian Res.* 182, 43–56. doi:10.1016/j.precamres.2010.06.020
- Jackson, S. E., Pearson, N. J., Griffin, W. L., and Belousova, E. A. (2004). The Application of Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry to *In Situ* U-Pb Zircon Geochronology. *Chem. Geology*. 211, 47–69. doi:10.1016/j.chemgeo.2004.06.017
- Liu, X., Gao, S., Diwu, C., Yuan, H., and Hu, Z. (2007). Simultaneous *In-Situ* Determination of U-Pb Age and Trace Elements in Zircon by LA-ICP-MS in 20  $\mu\text{m}$  Spot Size. *Chin. Sci. Bull.* 52, 1257–1264. doi:10.1007/s11434-007-0160-x
- Liu, X., Li, S., Li, X., Zhao, S., Wang, T., Yu, S., et al. (2018). Detrital Zircon U-Pb Geochronology and Provenance of the Sanxiatian Formation (Huade Group) in the North China Craton: Implications for the Breakup of the Columbia Supercontinent. *Precambrian Res.* 310, 305–319. doi:10.1016/j.precamres.2018.02.006
- Liu, Y., Gao, S., Hu, Z., Gao, C., Zong, K., and Wang, D. (2010). Continental and Oceanic Crust Recycling-Induced Melt-Peridotite Interactions in the Trans-North China Orogen: U-Pb Dating, Hf Isotopes and Trace Elements in Zircons from Mantle Xenoliths. *J. Petrol.* 51, 537–571. doi:10.1093/petrology/egp082
- Liu, Y., Hu, Z., Gao, S., Günther, D., Xu, J., Gao, C., et al. (2008). *In Situ* analysis of Major and Trace Elements of Anhydrous Minerals by LA-ICP-MS Without Applying an Internal Standard. *Chem. Geology*. 257, 34–43. doi:10.1016/j.chemgeo.2008.08.004
- Ludwig, K. R. (2003). *User's Manual for Isoplot 3.00: A Geochronological Toolkit for Microsoft Excel*. Berkeley, CA: Berkeley Geochronology Center.
- Vermeesch, P. (2012). On the Visualisation of Detrital Age Distributions. *Chem. Geology*. 312–313, 190–194. doi:10.1016/j.chemgeo.2012.04.021
- Wiedenbeck, M., Allé, P., Corfu, F., Griffin, W. L., Meier, M., Oberli, F., et al. (1995). Three Natural Zircon Standards for U-Th-Pb, Lu-Hf, Trace Element and Re Analyses. *Geostandards Newsl.* 19, 1–23. doi:10.1111/j.1751-908X.1995.tb00147.x
- Yuan, H. L., Gao, S., Dai, M. N., Zong, C. L., Gunther, D., Fontaine, G. H., et al. (2008). Simultaneous Determination of U-Pb Age, Hf Isotopes and Trace Element Composition of Zircon by Excimer Laser-Ablation Quadrupole and Multiple-Collector ICP-MS. *Chem. Geology*. 247 (1), 100–118. doi:10.1016/j.chemgeo.2007.10.003
- Zhai, M., Hu, B., Zhao, T., Peng, P., and Meng, Q. (2015). Late Paleoproterozoic-Neoproterozoic Multi-Rifting Events in the North China Craton and Their Geological Significance: A Study advance and Review. *Tectonophysics*. 662, 153–166. doi:10.1016/j.tecto.2015.01.019
- Zhao, G., He, Y., and Sun, M. (2009). The Xiong'er Volcanic belt at the Southern Margin of the North China Craton: Petrographic and Geochemical Evidence for its Outboard Position in the Paleo-Mesoproterozoic Columbia Supercontinent. *Gondwana Res.* 16, 170–181. doi:10.1016/j.gr.2009.02.004
- Zhao, G., and Zhai, M. (2013). Lithotectonic Elements of Precambrian Basement in the North China Craton: Review and Tectonic Implications. *Gondwana Res.* 23, 1207–1240. doi:10.1016/j.gr.2012.08.016
- Zhao, T. P., Zhai, M. G., Xia, B., Li, H., Zhang, Y. X., and Wan, Y. S. (2004). The SHRIMP Geochronology on the Zircons of the Xiong'er Volcanics: Constraints on the Initial Time of the Cover for North China Craton. *Chin. Sci. Bull.* 22, 2342–2349. doi:10.1007/BF03183721

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