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Mesoproterozoic age for Xiamaling Formation in North China Plate indicated by zircon SHRIMP dating

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Zircon grains of magmatic origin from tuffite layers in the Xiamaling Formation at Zhaojiashan Village, Xuanhua area, Hebei Province, were used for zircon dating with a Sensitive High-Resolution Ion Microprobe (SHRIMP II), which gives a weighted mean ²⁰⁷Pb/²⁰⁶Pb age of 1366±9 Ma. It shows a very similar age (1368±12 Ma) as the zircon dating from the Xiamaling Formation in Western Hill, Beijing. This age proposes that the Xiamaling Formation in the North China plate should be of Mesoproterozoic, instead of Neoproterozoic based on K-Ar, Ar-Ar dating. The new zircon age also indicates the development of macrofossils algae from the Mesoproterozoic age.

Xiamaling Formation, SHRIMP, zircon dating, Mesoproterozoic, North China Plate

The Xiamaling Formation, which is composed of clastic and carbonate rocks with a shallowing-up sequence, is developed between "Qinyu Movement^[1]," above and "Weixian Movement^[2]," below. It consists of four members, the third and fourth members of which are organicrich black shale or kuchersite. The Xiamaling Formation was considered as of Neoproterozoic based on its abundance of benthic macrofossil algae assemblages. The zircon dating proposes for the Xiamaling Formation a new position in Mesoproterozoic chronostratigraphy. This gives a new idea about the origin and evolution of macrofossils algae, and stratigraphic correlation, paleogeographic division and mapping of Meso-Neoproterozoic in the whole North China as well.

The Xiamaling Formation was named by Yel^[3] in its type section at Xiamaling Village, Mentougou District, Beijing. It consists of siltstone and shale interbedded with dolomite developed between siliceous rocks above and pebbly-sandstone rocks below. Kao^[4] used the Xiamaling Formation in the Meso-Neoproterozoic section in the Jixian area, and put the Xiamaling and Jing'eryu formations into the Qingbaikou System, Neoproterozoic. Later, Qiao^[1] reported "Qinyu Movement" in Qinyu,

Western Hill, Beijing based on a weathering crust developed on top of the Tieling Formation. The Qingbaikou System was considered as between 1000 and 800 Ma by the glauconite dating using K-Ar and Ar-Ar methods^[5-7].

In 2007, three layers of tuffaceous beds (5-8 cm thick) interbedded in black shale were first found by the authors from in the middle part of the Xiamaling Formation in Western Hill, Beijing. A large number of zircon grains of magmatic origin are gained from the tuffite. A SHRIMP age was first obtained from 19 of the zircon grains, yielding a concordia age (weighed average age) of $1368\pm12 \text{ Ma}^{[8]}$. This age challenges the traditional idea that the Xiamaling Formation belongs to Neoproterozoic, and propose a re-division and correlation of late Precambrian in the North China plate^[9]. This achievement draws attention and got support at the third workshop of the National Commission on Stratigraphy of China (NCSC) and China Geological Survey (CGS),

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Figure 1 Meso-Neoproterozoic column and contrast of the Xiamaling Formation (modified from Qiao^[9] and Gao^[8]).

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held in 2007. Later, another ten tuffaceous layers are distinguished by the authors at Zhaojiashan Village, Xuanhua area, Hebei Province.

A large number of zircon grains are gained again from the tuff. 16 zircon grains are used for ²⁰⁷Pb/²⁰⁶Pb aging, using a Sensitive High-Resolution Ion Microprobe (SHRIMP II) at Beijing SHRIMP Center. The new age, (weighed mean ²⁰⁷Pb/²⁰⁶Pb) 1366±9 Ma, is equal to that from the Xiamaling Formation (type section) in Western Hill, Beijing. The new evidence shows that the tuffaceous bed is widely dispersed in the North China plate. The new zircon dating of the Xiamaling Formation proposes a new age of macrofossils algae development in Mesoproterozoic, which is about 4.5 Ga earlier than formerly supposed.

1 Strata succession and tuffite locality

The Xiamaling Formation, developed in the northeast part of the North China plate, is at the top part of the Mesoproterozoic strata. It disconformably overlies on stramotolitic carbonate of the Tieling Formation and underlies the Luotuoling Formation (Figure 1). It is well exposed in the Yanshan area and the northern part of Taihang Range. The sedimentary center (the thickest) of the Xiamaling Formation is at Huailai and Xinglong area, where it consists of grey, grey-green, purple red and grey black silty shale and page-like siltstone, with a large amount of slate sandy lens in the lower part and lenticular muddy limestone with stramotolites in the upper part, with a thickness of 133–537 m.

At the Zhaojiashan section (Figure 2), where zircon



Figure 2 Mesoproterozoic strata in Xuanhua, Hebei Province and the sampling locality. Ch*c-d*, Changcheng Changzhougou-Dahongyu formations; Ch*d*, Changcheng Dahongyu Formation; Chg, Changcheng Gaoyuzhuang Formation; Jx, Jixian System; Jxy, Jixian Yangzhuang Formation; Jxw, Jixian Wumishan Formation; Jxh, Jixian Hongshuizhuang Formation; Qb, Qiangbaikou System; \in , Cambrian; O, Ordovician; C, Carboniferous; J, Jurassic; P, Permian; K, Cretaceous; E, Neogene.

samples were collected, the Xiamaling Formation has a thickness of 530 m, with its 4 members exposed. The first member is page-like sandstone interbedded with amygdaloidal structures of basalt. The second member is composed of thin bedding of sandstone with Bouma sequence and bentonite. The third member is black and grey black silty with ten layers of bentonite. The fourth member is grey and grey-green silty with thin sandy lens and a large number of limestone developed in the upper part. The underlying strata are the Mesoproterozoic Tieling Formation with the well-known "Qinyu Movement" between the two formations. The Neoproterozoic Luotuoling Formation overlies the Xiamaling Formation.

Samples (T07421-5) were collected from the middle part of the Xiamaling Formation in the Zhaojiashan section, Xuanhua area, Hebei Province. Sampling place is at Zhaojiashan Village (N 40°28'39"; E115°23'22"), 4 km northeast of Xinbao'an. The samples are distributed in 10 tuffite layers with 10-30 cm thickness totally. The tuffite is composed of illite, montmorillonite and mixture with micro-crystal feldspar, quartz and biotite, which reflects that the source rock belongs to neutral acid felsic magma, with geochemical character of K-rich element. Ash bed resolved in water is K-bentonite.

2 Analysis method

The SHRIMP analyses were performed at the Beijing SHRIMP Center, Chinese Academy of Geological Sciences, Ministry of Land and Resources of the People's Republic of China. The analysis procedures follow Liu and Lance^[11,12]. Mass resolution is ca. 5000 (1% peak height). A primary ion beam of ca. 4 nA, 10 kV O_2^- and ca. 40 µm spot diameter were used. The background was

measured on a single electron multiplier by cyclic stepping of the magnetic field, recording the mean ion counts of every five scans. For the zircon analyses, 35 ion species of Zr_2O^+ , ${}^{204}Pb^+$, ${}^{206}Pb^+$, ${}^{207}Pb^+$, ${}^{208}Pb^+$, ${}^{238}U^+$, $^{232}\text{Th}^{16}\text{O}^+$ and $^{238}\text{U}^{16}\text{O}^+$ were used. Interelement fractionation in ion emission of zircon was collected relative to the ANURSES references (TEM and SL13). TEM have U-Pb Concordia age, and ²⁰⁷Pb/²⁰⁶Pb is 416.8±1.1 Ma^[13,14], the content of U, Th and Pb is not homogeneous. SL13 is 572 Ma and the content of ²³⁸U processes 238 ppm. The software attached ISOPLOT and Squid, which was written by Prof. Ludwig, was used for data processing and concordia diagram^[15]. Measure ²⁰⁴Pb is used for common Pb correction. The result of model calculating used composition of common Pb supplied by Stacey and Kramers^[16]. The range of age error is limited within 1σ for all spots and more and the range of isotopic error is limited within 1σ for all spots. Therefore, yielding a weighted mean ²⁰⁷Pb/²⁰⁶Pb age has 95% confidence.

3 Analysis result

Zircon character of CL: On the bases of the Cathodoluminesecence (CL) images and to sharp contrast of feature in perspective light, the spot zircons are selected without crackle and inclusion. Zircons in T07421-5 are light yellow color and transparent crystals in column, simicone and subaround. Diameter of gains is between $100-200 \mu m$. Rationmeter of length and width of column crystal is 2:1-3:1, Figure 3 shows that the CL images of the zircons are the magmatic zoning and rhythms structure. Therefore, they are magma crystals. Content of Th, U and value of Th/U in zircon: Zircon



Figure 3 CL images of zircon grains from ash bedding of the Xiamaling Formation at Zhaojiashan, Xuanhua area.

illustrates high contents of Th and U in magma zircon and large value scale of Th/U ratio (usually larger than 0.4). The value of Th ranges from 23×10^{-6} to 84×10^{-6} in 16 gains of bentonite of the Xiamaling Formation (T07421-5), some shows high level. The value of U between 65×10^{-6} and 211×10^{-6} ; Th/U is among 0.33 -0.49 (Table 1). Testing those spots is mainly in zones of magma.

Zircon age: Totally 16 gains were sampled from tuffite (sample No. T07421-5) of the Xiamaling Formation. 3 spots (5.1, 14.1, 16.1) were different from others and not located near the concordia, suggesting that 14.1 must had suffered Pb's loss, with the majority 10 spots (1.1, 2.1, 3.1, 4.1, 6.1, 7.1, 8.1, 9.1, 11.1, 12.1, 13.1, 15.1) falling on the same concordia spots (Figure 4). These spots yield a weighed mean ²⁰⁷Pb/²⁰⁶Pb age of 1366±9 Ma, corresponding to MSWD = 1.44; All of the spots vield a weighed mean ²⁰⁷Pb/²⁰⁶Pb age of 1368±26 Ma, and then fourteen spots (except 5.1, 14.1) yield a weighed mean ²⁰⁷Pb/²⁰⁶Pb age of 1369±11 Ma. Therefore, a weighed mean ²⁰⁷Pb/²⁰⁶Pb age of 1366±9Ma (thirteen spots) indicates the forming age of zircon and tuffite.

4 Discussion

Based on zircon CL (Figure 3) and features of magma zircons from the value of Th/U (Table 1), an age of



Figure 4 U-Pb concordia diagram for zircon in sample T07421-5.

1366±9 Ma (MSWD=1.44) is gained for the tuffite layers in the Xiamaling Formation, at Zhaojiashan Village, Xuanhua area, Hebei Province. However, the age given by previous study has caused an unsolved problem in stratigraphic division, correlation and tectonic patter in the Meso-Neoproterozoic strata in the North China plate.

New division and correlation in Meso-Neoproterozoic: the Xiamaling Formation was traditionally considered as belonging to Neoproterozoic based on Ar-Ar age from the Tieling and Luotuoling Formations (<1000 Ma). The present zircon dating proposes a new geochronological age for the Xiamaling Formation in the North

| Table 1 | U-Pb isotopic compositions of zircons from sample T07421-5 | | | | | | | | | | | |
|---------|------------------------------------------------------------|-------------|-------------|--------------------|--------------------|------------|--------------------------------------|---------------|----------------------------------------|-----------------|-------------------|-------|
| Spot | ²⁰⁶ Pbc | U | Th | ²³² Th | ²⁰⁶ Pb* | 206Pb/238U | ²⁰⁷ Pb/ ²⁰⁶ Pb | Non-concordor | ²⁰⁷ Pb*/ ²⁰⁶ Pb* | 207Pb*/235U | 206Pb*/238U | Error |
| | (%) | (10^{-6}) | (10^{-6}) | / ²³⁸ U | (10^{-6}) | Ma | Ma | (%) | ±% | ±% | ±% | |
| 1.1 | 0.40 | 146 | 69 | 0.49 | 29.9 | 1377±28 | 1371±35 | 0 | 0.0875 ± 1.8 | 2.872 ± 2.9 | 0.2381±2.3 | 0.775 |
| 2.1 | 0.40 | 106 | 39 | 0.38 | 21.6 | 1366±15 | 1388±68 | 2 | 0.0882 ± 3.5 | 2.870 ± 3.8 | $0.2360{\pm}1.2$ | 0.328 |
| 3.1 | 0.98 | 97 | 35 | 0.38 | 19.5 | 1345±16 | 1350±79 | 0 | 0.0865 ± 4.1 | 2.770±4.3 | 0.2319±1.3 | 0.309 |
| 4.1 | 0.82 | 93 | 35 | 0.39 | 18.7 | 1343±17 | 1365±59 | 2 | 0.0872 ± 3.1 | 2.785±3.4 | 0.2317 ± 1.4 | 0.414 |
| 5.1 | 0.46 | 119 | 45 | 0.40 | 22.8 | 1296±13 | 1362±41 | 5 | 0.0871 ± 2.1 | 2.672 ± 2.4 | $0.2226{\pm}1.1$ | 0.461 |
| 6.1 | 0.62 | 172 | 84 | 0.51 | 35.1 | 1365±12 | 1343±47 | -2 | 0.0862 ± 2.4 | $2.804{\pm}2.6$ | 0.2359 ± 0.94 | 0.361 |
| 7.1 | 1.01 | 78 | 25 | 0.33 | 15.7 | 1348±18 | 1392±75 | 3 | 0.0884 ± 3.9 | $2.840{\pm}4.2$ | $0.2326{\pm}1.5$ | 0.354 |
| 8.1 | 0.30 | 113 | 41 | 0.38 | 22.8 | 1358±21 | 1392±70 | 3 | 0.0884 ± 3.7 | $2.860{\pm}4.0$ | 0.2345±1.7 | 0.422 |
| 9.1 | 0.85 | 127 | 56 | 0.45 | 26.4 | 1393±15 | 1385±74 | -1 | 0.0881 ± 3.8 | $2.930{\pm}4.0$ | 0.2411 ± 1.2 | 0.290 |
| 10.1 | 0.88 | 65 | 23 | 0.37 | 13.3 | 1373±32 | 1394±71 | 2 | 0.0885 ± 3.7 | $2.900{\pm}4.5$ | 0.2374 ± 2.5 | 0.565 |
| 11.1 | 1.18 | 86 | 39 | 0.47 | 16.4 | 1280±49 | 1379±75 | 8 | 0.0878 ± 3.9 | 2.660 ± 5.8 | 0.2196 ± 4.2 | 0.734 |
| 12.1 | 0.63 | 116 | 44 | 0.39 | 23.7 | 1370±14 | 1398±47 | 2 | 0.0887 ± 2.4 | 2.896 ± 2.7 | 0.2367±1.1 | 0.425 |
| 13.1 | 0.29 | 140 | 63 | 0.47 | 28.9 | 1385±13 | 1358±34 | -2 | 0.0869±1.8 | 2.871±2.0 | 0.2396±1.0 | 0.506 |
| 14.1 | 0.86 | 211 | 138 | 0.68 | 32.6 | 1056±14 | 1338±52 | 27 | 0.0860 ± 2.7 | 2.111±3.1 | $0.1780{\pm}1.5$ | 0.476 |
| 15.1 | 0.61 | 102 | 40 | 0.41 | 20.8 | 1373±18 | 1359±68 | -1 | 0.0869±3.5 | 2.840±3.8 | 0.2373±1.4 | 0.373 |
| 16.1 | 0.52 | 143 | 58 | 0.42 | 30.3 | 1415±26 | 1365±41 | -3 | 0.0872 ± 2.1 | 2.951±2.9 | 0.2454±2.0 | 0.689 |

Errors are 1 σ , Pbc and Pb*indicate the common and radiogenic portions, respectively. Error in standard calibration was 0.60%, common Pb corrected using measured 204Pb.

China plate. However, the new SHRIMP dating was put forward some proposals: (1) the Xiamaling Formation in the Xuanhua area, Hebei Province, should be of Mesoproterozoic, and the Xiamaling Formation (the type section) at Western Hill, Beijing should have similar age. The "Qinyu movement" on top of the Xiamaling Formation and the "Weixian Movement" overlays the Xiamaling Formation should have a range from 1400 Ma to 1200 Ma; and (2) The new age proposes a big sedimentary break between the Mesoproterozoic and Neoproterozoic chronostratigraphic column in the North China plate. The lost strata of this break may exist above the Xiong'er Group^[9] in the southern margin of the North China plate or in the Kunyang Group in the Yangtze plate^[13]. Moreover, we need to determine the late Precambrian strata, which are limited by an age of diabase dikes (924±8 Ma^[19]) in Jiao-Liao-Xu-Huai Sub-province.

Biota evolution succession: A great number of macrofossil algae are found in the third and the fourth layers in shale with thin bedding limestone, gained from the Xiamaling Formation, Zhaojiashan, Xuanhua area, Hebei Province^[20]. The macrofossil algae were contrasted with the Neoproterozoic biota assemblage on its quantity, feature and an old Ar-Ar age. But the question is that different algae forms could not be classified as one assemblage and the new age (1366 \pm 9 Ma) puts the formation forward to Mesoproterozoic and makes biota evolution advance to 4.5 Ga that becomes new evidence

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for global biota evolution.

Tectonic significance: Qiao found red soil weathering crust at top of the Tieling Formation and named it "Qinyu movement"^[1], which was correlated with the Rodinia. The result of the SHRIMP age of the Xiamaling Formation indicates that (1) The new age of the Xiamaling Formation has an effect on original locality in strata column, not only it moved forwards to Mesoproterozoic, but also it represents the sedimentary record during a new cycle of tectonic extension. Because the Luotuoling Formation widely overlies the Xiamaling Formation in the North China plate, we need to recognize a suit of clastic rocks in the southern margin of the North China plate and in Jiao-Liao-Xu-Huai Subprovince. The location of the clastic rocks is important evidence for the column. (2) The "Qinyu Movement" may occur at 1400 Ma, which represents an uplift movement of the Sino-Korean Plate after Yan-Liao Aulacogen, and corresponds to the time of the main breakup of Columbia Supercontinent^[21-25]. Therefore, the "Qinyu Movement" is the expression of the breakup of Columbia Supercontinent.

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