Phase Equilibria Modeling and Zircon Dating for Precambrian Metapelites from the Xinghuadukou Complex in the Lulin Forest of the Erguna Massif, Northeast China

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Phase equilibria modeling and zircon dating for Precambrian metapelites from the Xinghuadukou Complex in the Lulin Forest of the Erguna Massif, Northeast China

Abstract

The nature and formation time of the Xinghuadukou Complex in Northeast China are important for determining the tectonic evolution of the Precambrian geological evolution of the Erguna Massif. In this study, we present the results of zircon U-Pb dating of two metapelites from the complex. Detrital and metamorphic zircons from the metapelites yield a depositional age of ~601 Ma and a metamorphic age of 496 to 509 Ma, indicating that the supracrustal rocks formed during the Neoproterozoic and recorded Pan-African metamorphic events in the Erguna Massif. Garnet porphyroblasts in Sil-Grt-Bt-Ms paragneiss show diffusion zoning, implying a decreasing *P-T* trend. Based on mineral transformation and *P-T* estimates using conventional geothermobarometers and phase equilibria modelling, three metamorphic stages were determined, including an early prograde metamorphic stage, a near peak upper amphibolite facies metamorphic stage, and a near-isobaric cooling stage with *P-T* conditions of 645°C and 6.1 kb, 711–722°C and 5–6 kb, and 625°C and 4.4 kb, respectively. The metamorphic history of the Xinghuadukou Complex is thus defined by a clockwise *P-T* trajectory, which implies that the metamorphism of the metapelites occurred in a post-collision tectonic setting.

1. Introduction

The Central Asian Orogenic belt (CAOB) is a giant accretionary orogeny among the Siberian Craton, the North China Craton and the Tarim Craton, which consists of a series of island arcs, forearc or back-arc basins, ophiolitic belts and microcontinents of Neoproterozoic to Mesozoic age (Sengör et al., 1993; Sengor and Natalin, 1996; Windley et al., 2007). This orogeny is one of the largest and the most complex accretionary collages responsible for considerable Phanerozoic juvenile crustal growth (Jahn et al., 2001; Jahn et al., 2000; Jahn, 2004; Kröner et al., 2010; Kröner et al., 2007; Rytsk et al., 2011; Safonova et al., 2011; Safonova and Santosh, 2014; Safonova et al., 2009; Turkina et al., 2007; Wu et al., 2000; Wu et al., 2003a; Wu et al., 2003b; Wu et al., 2005a; Wu et al., 2002; Wu et al., 2011; Xiao and Santosh, 2014). The prolonged accretionary processes recorded the breakup of the Rodinia Supercontinent and the creation of the Eurasian Supercontinent (Dobretsov et al., 2003; Li et al., 2008; Torsvik and Cocks, 2004). Northeast China, tectonically termed the Xing'an-Mongolian Orogenic Belt (XMOB), is in the eastern segment of the CAO (Jahn, 2004; Li, 2006; Sengör et al., 1993; Sengor and Natalin, 1996; Windley et al., 2007; Wu et al., 2011). This belt has been traditionally subdivided into a series of massifs, including the Erguna Massif in the northwest, the Xing'an and Songliao massifs in the central area, and the Jiamusi and Khanka massifs in the east, separated by major faults (**Fig. 1a**). The tectonic evolution of NE China was closely related to the Paleo-Asian Ocean and Paleo-Pacific Ocean regimes during the Paleozoic–Early Mesozoic (Han et al., 2012; Li, 2006; Liu et al., 2017; Windley et al., 2007), and related to the western Pacific Ocean and Mongol-Okhotsk Ocean regimes during and following the

Mesozoic (Meng et al., 2011; Meng et al., 2010; Tang et al., 2015; Wang et al., 2012; Wu et al., 2011; Xu et al., 2013). Despite general agreement on the geological evolution of NE China from the Paleozoic to the Mesozoic, the Precambrian history of this area remains controversial because of the limited research on the Precambrian units of NE China. Previous studies have identified numerous Precambrian terranes within microcontinental massifs in NE China, as demonstrated by field relationships and zircon data (Ge et al., 2015; 1993; 1993; Miao et al., 2007; Tang et al., 2013; Wu et al., 2012; Xu et al., 2015; Zhao et al., 2016a; Zhao et al., 2016b; Zhao et al., 2016c). However, only a small number of studies have been undertaken on these Precambrian rocks and the limited distribution of these units hinders a greater understanding of the Precambrian tectonic evolution of NE China.

In this paper, we focus on the Xinghuadukou Complex, a unit that has been interpreted to be the oldest stratigraphic sequence in the Erguna Massif. We investigate meta-sedimentary rocks from the complex and present several new LA-ICP-MS zircon U-Pb, petrological and mineralogical chemical data, as well as phase equilibria modeling, to constrain the timing of metamorphism, *P-T* conditions and the *P-T* path of the Xinghuadukou Complex. The results provide important insights into the formation, metamorphism and tectonic evolution of the Xinghuadukou Complex.

2. Geological Setting

The Erguna Massif is a significant tectonic unit bounded by the Mesozoic Mongolia-Okhotsk Suture to the northwest and the Paleozoic Xiguitu-Xiguitu Suture to the southeast (**Fig. 1b**). This massif was traditionally considered to be composed of Proterozoic to Paleozoic volcano-sedimentary strata and granites (HBGMR, 1993; IMBGMR, 1993) with a Precambrian metamorphic basement, primarily outcropping in the northwest part of the massif. In this location, widely developed thick Mesozoic volcanic rocks cover the middle and southern part of the Erguna Massif. Recent geochronological data found in the Erguna Massif indicate that this area underwent multiple stages of granitic magmatism, with the majority of intrusions being emplaced during the Cretaceous (132–118 Ma), Jurassic to Triassic (220–182 Ma) and Early Paleozoic (517–416 Ma), with lesser magmatism during the Late Paleozoic (336–224 Ma), Neoproterozoic (927–737 Ma), Paleoproterozoic (1854–1837 Ma) and Nearchean (2600 Ma) (Ge et al., 2015; Shao et al., 2015; Sun et al., 2013b; Tang et al., 2013; Wu et al., 2011; Zhao et al., 2016b; Zhao et al., 2016c). The Erguna Massif consists of Neoproterozoic [608–949 Ma] meta-sediments (Xinghuadukou Complex, Erguna Group, Jiageda Group) and a Late Precambrian sequence (Mohe Complex) (Miao et al., 2007; Wu et al., 2012; Zhou et al., 2011a; Zhou et al., 2011b), in which khondalites record a Pan-African (~500 Ma) metamorphic event (Zhou et al., 2011b).

As the metamorphic basement of the Erguna Massif, the Xinghuadukou Complex is primarily distributed in areas around Mohe, Tahe, Huzhong, Huma, Xinlin and Qiqian, (Biao et al., 1999; Ge et al., 2015; Miao et al., 2007; Shanghu and Zheng, 2012; Wu et al., 2012; Zhou et al., 2011a) (**Fig. 1b**). It is comprised of amphibolites, schists, leptynites, leucoleptynites, marbles, gneisses and migmatites. Recent research suggests that the original Xinghuadukou Complex should be divided into meta-granitic rocks and meta-supracrustal rocks, and that the Xinghuadukou Complex should be limited to the term of meta-supracrustal rocks (Biao et al., 1999; Ge et al., 2015; Liu et al., 2017; Shanghu and Zheng, 2012). These meta-supracrustal rocks series are a typical volcano-sedimentary formation including meta-basic and meta-acidic volcanic rocks and meta-sedimentary rocks. These

meta-supracrustal rocks are scattered as xenoliths in the meta-granitic plutons of different shapes and sizes because of the effect of multiple granitic magmatic events and structural deformation.

3. Analytical Methods

Two representative samples were selected for detailed study. The samples were used for whole rock analyses as well as for determining the chemical composition of minerals by electron microprobe. Fresh whole-rock samples were selected to grind in an agate mill to less than 200 mesh in size. The major elements of the samples were analyzed on fused glass disks using a PHILIPS-2404 X-ray fluorescence spectrometer at the Tianjian Institute of Geology and Mineral Resources, China. The analytical results of the major elements in the Xinghuadukou Complex are listed in **Table 1**.

The compositions of minerals in thin section were analyzed with an electron microprobe analyzer (EMPA; JXA-8200, JEOL) with five wavelength-dispersive spectrometers using synthetic and natural minerals, glass, and pure oxides as standards at the Laboratory of Jilin University, China. The concentrations of Na, Mg, Al, Si, K, Ca, Ti, Cr, Mn and Fe in these minerals were determined. Counting times were 20 s at the peak and on the background. The applied acceleration voltage and beam current were 15 kV and 10 nA, respectively. The beam diameter for all analyses was typically 2–5 μm .

For the zircon dating, zircons were separated using standard heavy liquids and magnetic methods and were handpicked under a binocular microscope. Purified zircon grains were then mounted in epoxy and polished to about half thickness to expose the cores of the grains. Zircons were photographed in transmitted and reflected light, and imaged using cathodoluminescence (CL). CL images were collected using a CL spectrometer (Garton Mono CL3+) equipped on a Quanta 200F ESEM with a 2-min scanning time at conditions of 15 kV and 120 nA. LA-ICP-MS zircon U-Pb isotopic analyses were performed using an Agilent 7500a inductively coupled plasma mass spectrometer (ICP-MS) equipped with 193-nm laser, housed at the Key Laboratory of Mineral Resources Evaluation in Northeast Asia, Ministry of Land and Resources, China. Zircon 91500 was used as an external standard for age and the standard silicate glass NIST SRM 610 was applied for instrument optimization. The concentrations of U, Th and Pb elements were calibrated using ^{29}Si as an internal calibrant. $^{207}\text{Pb}/^{206}\text{Pb}$, $^{206}\text{Pb}/^{238}\text{U}$ and $^{207}\text{Pb}/^{235}\text{U}$ ratios and apparent ages were calculated using ICPMSDataCal Ver.6.7 (Liu et al., 2010; Liu et al., 2008). The age calculations and Concordia plots were made using Isoplot (ver.3.0) (Ludwig, 2003).

4. Sample Description and Mineral Compositions

4.1. Description of the studied samples

Along the road from Tahe to the Lulin Forest, where the Xinghuadukou Complex outcrops (**Fig. 2**), rock samples were taken a zone of the supracrustal Xinghuadukou Complex. The outcrop shows that the rock had experienced partial melting with evidence of small-scale leucosome bands and migmatite (**Fig. 3**).

Based on thin-section study using a polarizing microscope the samples can be characterized as follows:

Sil-Grt-Bt-Ms paragneisses (Sample Z1545-5, Z1545-9, Z1545-10, and Z1545-11) for this study contain garnet porphyroblasts in a matrix consisting of sillimanite, quartz, biotite, muscovite and plagioclase with sericitic alteration. The rock shows a foliation defined by oriented sillimanite and biotite (**Fig. 4**). The grain size distribution of the garnet is bimodal. A small number of several-mm-

sized garnets occur, but several 0.2-mm-sized relic garnet grains are resorbed by muscovite and biotite. Garnet porphyroblasts are generally xenomorphic and in contact with biotite, quartz and sillimanite. Certain mm-sized garnet grains are nearly free of inclusions, whereas inclusions are common in the smaller garnet grains. The inclusions in the garnet are commonly biotite and quartz. There are three different types of biotite. The first type occurs as 0.2-mm-size inclusions of garnet with a reddish-brown color without obvious cleavage, and the second is a sheet type of reddish-brown biotite with rutile inclusions in a matrix along with tablet and fibrous sillimanite. The third type is the common brown biotite ranging from 0.2 to 1 mm in size with obvious cleavage oriented along the dominant foliation. Sillimanite occurs as a tablet and fibrous shape ranging from 0.05 to 0.5 mm in size. Sillimanite can be divided into four occurrences: (1) as a matrix mineral in the host rock, (2) distributed along the cleavage of biotite, (3) as inclusions in late muscovite and quartz, and (4) as inclusions in garnet. Muscovite occurs as a coarse-to-medium grained sheet crystal crosscutting the dominant foliation, distributed around relic garnet porphyroblasts and the core of selvages of biotite and sillimanite. Plagioclase has sericitic alteration. Quartz occurs as coarse-to-medium-grained rounded or elongated crystals in the matrix. Accessory phase minerals are rutile, monazite, zircon and zoisite. Based on textural observation and the mineral compositions previously presented, the observed peak assemblage is considered to contain garnet, sillimanite, quartz, biotite with or without ilmenite, and muscovite and plagioclase in matrix should be a retrograde phase. The list of main minerals and their modal proportions (vol. %) are listed in **Table 2**.

4.2. Chemical compositions of the minerals

Spot analyses using EMP were undertaken in all chemical domains of garnet to show the compositional change in garnet in the studied rocks during metamorphism. In general, garnet is rich in an almandine component showing weak core-to-rim compositional zonation (**Fig.5**). The relative end-number components vary from 72 to 77 mol% in almandine, 11 to 17 mol% in spessartine, 7 to 10 mol% in pyrope and 2 to 3 mol% in grossular (see **Table 3**).

The biotites are dominated by ferribiotite with FeO^T and MgO contents ranging from 19.25 to 22.89 wt.% and 5.64 to 9.39 wt.%, respectively. The TiO_2 contents vary from 0.28 to 3.99 wt.%. The matrix biotite shows lower X_{Mg} of 0.31–0.33 than that as inclusions in garnet ($X_{\text{Mg}}=0.37\text{--}0.46$). The significant negative correlation between FeO^T and MgO contents is the result of Fe-Mg exchange during metamorphism.

Muscovite occurs in the matrix around relic garnet, with Si ranging from 3.06 to 3.07 p.f.u. (**Table 3**) with a relative high value of X_{Mg} ($X_{\text{Mg}}=0.42\text{--}0.44$).

5. Metamorphic *P-T* Conditions

5.1. Methods

Metamorphic *P-T* conditions for the metapelites from the Xinghuadukou Complex were calculated from both traditional geothermobarometry and pseudosections. On the basis of the internally consistent thermodynamic database, the garnet-biotite (GB) (Holdaway, 2000) geothermometer, Ti-in-biotite geothermometer (Wu and Chen, 2015) and garnet-biotite-muscovite-aluminosilicate-quartz (GBMAQ) geobarometer (Wu and Zhao, 2007) were applied to calculate *P-T* conditions.

P-T section of the Sil-Grt-Bt-Ms paragneisses was carried out using Gibbs energy minimization (Connolly, 2005) with the thermodynamic database of Holland and Powell (1998, as revised in 2003) (Holland and Powell, 1998). Garnet- and melt-solution models were used from White *et al.* (2007) (White *et al.*, 2007), and biotite-solution model from Tajčmanová *et al.* (2009b) (Tajčmanová *et al.*,

2009). The models of Newton *et al.* (1980) for plagioclase and Thompson and Hovis *et al.* (1979) (Thompson and Hovis, 1979) for K-feldspar were used. Mn solution in garnet and staurolite was accounted for by the Mn end-members introduced by Tinkham *et al.* (2003) (Tinkham *et al.*, 2003). Based on the dominant minerals and their chemical compositions, the phase equilibria of the Xinghuadukou Complex metapelites from the Lulin Forest can be modeled in the system MnNCKFMASH (MnO-Na₂O-CaO-K₂O-FeO-MgO-Al₂O₃-SiO₂-H₂O) for the *P-T* ranges from 0.3 to 1.0 GPa and 500 to 800 °C. Although the Mn content of these rocks is low, MnO was included in the modeling because of its influence on the stability of garnet at low temperatures (Spear, 1995; Tinkham *et al.*, 2003). The effect of Fe³⁺ is also neglected in this study, because although potentially important, no magnetite was present in the studied samples, and the amount of Fe³⁺ in biotite and garnet was thus assumed to be insignificant. The bulk composition was obtained using X-ray fluorescence (XRF). The resulting bulk-rock compositions (in mol%) used for calculation are indicated in the upper left inset of the calculated *P-T* phase diagram section (**Fig. 6**).

5.2. Results

On the basis of the mineral assemblage of the metapelite of the Xinghuadukou Complex, the peak mineral assemblage should be Bt + Grt + Sil + Qtz, and the retrograde phase Bt + Mus + Grt + Qtz ± Pl. From evidence of surrounding migmatites with leucosome, K-feldspar from partial melting (Fig.) and late, crosscutting muscovite in the cores of biotite and sillimanite, this rock should have experienced fluid-saturated solidus melting, muscovite dehydration melting, and partial biotite dehydration melting reactions. This finding means that the muscovite in the metapelite was consumed during prograde metamorphism. The stable field should be in the area of Bt + Grt + Kfs + Sil + liq (**Fig. 6**). The metamorphic reaction within this area along the isobaric heating path is Bt + Sil + Qtz = Gt + Kfs + liq. Garnet was once regarded to be one of the most resistant minerals to diffusion during most metamorphism. However, it has been recognized that diffusion of divalent cations is capable of relaxing the compositional zoning in garnet at temperatures reached during amphibolite facies metamorphism (Florence and Spear, 1991); thus, Fe-Mg minerals, such as garnet and biotite, generally do not record their peak metamorphic compositions. In this case, the biotite inclusions of garnet will likely preserve the prograde metamorphism or near-peak metamorphic conditions. The X_{Mg} and Ti in biotite increase with an increase in temperature. We obtained two different high X_{Mg} values (0.37 and 0.46) for biotite inclusions in garnet. However, the latter one with the higher X_{Mg} value has notably little Ti, showing the latter biotite is a prograde product during the growth of the garnet. The former one with an X_{Mg} value of 0.37 and 0.21 p.f.u. of Ti can be regarded as the near-peak T condition of metamorphism. Based on the isopleth of the X_{Mg} of biotite, X_{Gr} of garnet and Ti-in-biotite geothermometer, the near-peak T conditions are 711–722 °C with a pressure ranging from 5–6 kb. The latter biotite should be at the *P-T* conditions of 645 °C and 6.1 kb as a prograde metamorphic record.

The observed retrograde assemblage Bt-Grt-Mus-Pl-Sil-Qtz defines a *P-T* range of 585–660 °C and 3.5–5.4 kb from phase equilibria modelling. At an average pressure of 4.5 kb, the measured X_{Py} and X_{Mg} of the garnet core isopleth yield a temperature and pressure of 645 °C and 4.5 kb, respectively, under solidus conditions. To constrain the retrograde *P-T* conditions, the relic garnet contacting the muscovite and biotite are chosen to apply to the GB geothermometer. The result yields a retrograde temperature of approximately 625 °C. GBMAQ geobarometer results show that the retrograde pressure is approximately 4.4 kb. In this case, a nearly isobaric cooling path is

suggested from the results.

For cooling along the path from point B to point C, the melt begins to crystallize as reactions of $\text{Grt} + \text{Kfs} + \text{liq} = \text{Bt} + \text{Sil}$. The crystallizing melt will release H_2O , continuously providing an internal source of H_2O for retrogression. Garnet will be consumed and biotite with sillimanite will be produced. It is unlikely that the exactly the same amounts of garnet grown from prograde metamorphism will be consumed as some parts of garnet will likely be preserved. The biotite and sillimanite produced forms selvages and are dispersed in the matrix with quartz (Spear et al., 1999; Wei, 2016). With continued cooling of the rock, it will encounter the retrograde of muscovite dehydration melting reaction, and enter the field of $\text{Bt-Mus-Grt-Kfs-Sil-Qtz-liq}$ with the reaction of $\text{Grt} + \text{Sil} + \text{Kfs} + \text{liq} = \text{Mus} + \text{Bt} + \text{Qtz}$. With the decrease in temperature, the rock continuously retrogrades and produces plagioclase with the reaction $\text{Grt} + \text{Sil} + \text{Kfs} + \text{liq} = \text{Mus} + \text{Bt} + \text{Pl} + \text{Qtz}$. The rock will continuously consume the garnet until the K-feldspar and melt totally disappear. If there is no melt-loss occurring, the rock will reach equilibrium without garnet present. However, if a degree of melt-loss occurs, the retrograde reactions will not reach equilibrium, and a fraction of relic garnet will be preserved. When melt-loss occurs, domains without sufficient garnet will have an assemblage of $\text{Bt-Mus-Sil-Qtz}(\pm)\text{Pl}$, and domains without sufficient sillimanite will have an assemblage of $\text{Bt-Mus-Grt-Qtz}(\pm)\text{Pl}$.

6. U-Pb Zircon Isotope

Zircons from sample Z1545-5 are colorless, transparent to translucent and prismatic, with well-defined prism faces and a subhedral shape (**Fig. 7**). The zircons range in size from 50 to 120 μm , with length to width ratios of 1:3. CL imaging reveals that a few large grains show complex internal structures, with irregular cores and narrow rims, indicating they are probably a result of overgrowth during late metamorphism or alteration. Generally, the grains consist of thin dark rims that are unzoned or show weak zoning from CL images, whereas most of the cores have well-defined oscillatory or planar zoning (**Fig. 7a**). Sixty-eight analyses were performed on 67 zircon grains, yielding ages ranging from a $^{206}\text{Pb}/^{238}\text{U}$ age of 479 Ma (spot Z1545-5-15) to a $^{207}\text{Pb}/^{206}\text{Pb}$ age of 2605 Ma (spot Z1545-5-10). The results are listed in **Table 4** and shown in **Fig. 8**. The majority of the 67 zircons from this sample plot near the Concordia curve barring eight analyses that record Pb loss. Four Neoproterozoic grains yield ages from 2509 to 2606 Ma with peak at 2518 Ma. Nine analyses of Paleoproterozoic zircon grains yield ages ranging from 1618 to 2343 Ma with three peaks at 1825 Ma, 2179 Ma, and 2351 Ma. Fourteen analyses of Mesoproterozoic zircon grains present ages ranging from 1011 to 1567 Ma with three peaks at 1012 Ma, 1390 Ma, and 1576 Ma. Thirty-four Neoproterozoic zircon grains yield ages from 592 to 999 Ma with the main peaks at 602 Ma, 698 Ma, 797 Ma, 848 Ma, and 917 Ma. The remaining six Cambrian zircons have ages from 479–530 Ma with peaks at 518 Ma.

Sample Z1545-10 contains a population of fine-grained, colorless to light yellow, transparent zircon crystals that are mostly prism or oval in shape. The zircon grains have lengths of 50–130 μm and length/width ratios of 1:3 (**Fig. 7b**). A total of 76 points were analyzed from 75 grains; the results are listed in **Table 4** and shown in **Fig. 8**. The data show a range of ages ranging from a $^{206}\text{Pb}/^{238}\text{U}$ age of 496 Ma (spot Z1545-10-06) to a $^{207}\text{Pb}/^{206}\text{Pb}$ age of 2695 Ma (spot Z1545-10-33). Overall, the ages can be roughly divided into five groups. One Neoproterozoic grain yields an age of 2695 Ma, the oldest recorded from the sample. Ten analyses of Paleoproterozoic zircon grains

present ages ranging from 1653 to 1965 Ma with peaks at 1660 Ma, 1841 Ma, 1915 Ma and 1978 Ma. Eighteen analyses of Mesoproterozoic zircon grains yield a range of ages from 1030 to 1525 Ma with peaks at 1250 Ma, 1360 Ma and 1488 Ma. Forty-six Neoproterozoic zircon analyses result in ages ranging from 758 to 992 Ma, with age peaks at 808 Ma, 901 Ma and 996 Ma. The younger Cambrian zircon ages of 496 Ma and 509 Ma with an obvious low Th/U ratio relative to the other grains in the sample indicate a metamorphic origin.

7. Discussion

7.1. Ages of the rocks from the Xinghuadukou Complex

The Xinghuadukou Complex was initially described as a sequence of Paleoproterozoic-to-Mesoproterozoic volcano-sedimentary rocks named the Xinghuadukou “Group” (Biao et al., 1999; 1993; Sun et al., 2002). However, this designation was based on lithostratigraphic relationships and unreliable isotopic ages (Biao et al., 1999; HBGMR, 1993; Shanghu and Zheng, 2012). In recent years, many new geochronological data by SHRIMP, LA-ICP-MS and single-grain U-Pb dating of zircons in the Erguna Massif have been published (Ge et al., 2005; Ge et al., 2015; Miao et al., 2007; Wang et al., 2017; Wu et al., 2012; Zhou et al., 2017; Zhou et al., 2011a). Miao (Miao et al., 2007) dated the Xinghuadukou Complex in the Xinlin-Hanjiayuanzi area using the U-Pb SHRIMP method and suggested that it formed during the Cambrian or Neoproterozoic. Zhou et al., (2011a) (Zhou et al., 2011a) dated detrital zircons from four metasedimentary rocks in the Mohe-Hongqi area using the LA-ICP-MS method. The results showed that the deposition of the sedimentary rocks from the Xinghuadukou Complex occurred no earlier than ~600 Ma and recorded the Pan-African metamorphic age of ~500 Ma. Wu et al. (2012) analyzed 96 detrital zircons using the U-Pb LA-ICP-MS method from the Xinghuadukou Complex in the Lulin Forest yielding ages concentrated from 749 Ma to 2765 Ma (igneous zircons) and 448–716 Ma (metamorphic zircons), suggesting that the sedimentary rocks from the Xinghuadukou Complex were deposited after 749 Ma. Ge et al. (2015) dated 12 samples (one is a meta-volcanic protolith) from the so-called “Xinghuadukou Group” with different ages at Mangui (957 Ma), Huma (~850 Ma), Hongqi Forest (844 Ma and 820 Ma), Qiqian (810 Ma and 418 Ma), Pangu (794 Ma and 786 Ma), Mohe (479 Ma and 265 Ma), and Lulin Forest (192 Ma and 209 Ma). Although this previously published data show the presence of the Precambrian basement of the Erguna Massif, the formation age of the supracrustal rocks of the Xinghuadukou Complex remains controversial.

As described in Section 3, the dated samples Z1455-5 and Z1545-10 are garnet-sillimanite gneisses, and their protolith is pelitic-sandy sedimentary rocks. Consequently, the zircons from the gneisses should be detrital or metamorphic in origin, and therefore their ages represent the ages of their provenances or metamorphism. The U-Pb dating results from Z1545-5 reveal a range of ages from 479 ± 8 Ma to 2605 ± 55 Ma with clusters at 2029–2606 Ma, 1263–1851 Ma, 909–1169 Ma, 849–850 Ma, 749–832 Ma, 640–731 Ma, 592–608 Ma, and 479–530 Ma. The youngest group, with ages ranging from 479 to 530 Ma and low Th/U ratios (0.03–0.36) with a weighted mean $^{206}\text{Pb}/^{238}\text{U}$ age of 500 ± 18 Ma (MSWD=2.8), and the zircon grains with analytical spots of Z1545-5-15, Z1545-5-14 and Z1545-5-12 (**Fig.7**), have distinguished core and rim structures, which indicate that they are of metamorphic origin. The zircons of the second youngest group of 592–608 Ma age are euhedral to subhedral and prismatic with clear oscillatory zoning and a high Th/U ratio (0.33–0.78), showing typical characteristics of igneous origin. Therefore, we interpret that the 592–608 Ma group

with a weighted mean age of 601 ± 12 Ma (MSWD=0.12) should define the maximum depositional age of the protolith of the garnet-sillimanite gneisses within the Xinghuadukou Complex. The dating results from Z1545-10 show a range of ages ranging from 496 ± 15 Ma to 2695 ± 16 Ma with clusters at 2695 Ma, 1835–1965 Ma, 1236–1653 Ma, 884–1030 Ma, 758–853 Ma, and 496–509 Ma. We suggest that the youngest zircon age group of 496–509 Ma with low Th/U ratios (0.13–0.14) and obvious core-rim structure is of metamorphic origin. The youngest detrital zircon of this sample, having an age of 758 ± 18 Ma, provides the maximum limit on the depositional age of the protolith of the garnet-sillimanite gneisses. In summary, our new geochronological data on zircons from these two samples indicate that the Xinghuadukou Complex in the Lulin Forest area was deposited during the Neoproterozoic (at least 601 Ma), rather than the Paleoproterozoic-to-Mesoproterozoic (Biao et al., 1999; HBGMR, 1993; Sun et al., 2002), and they recorded the time of metamorphism as Pan-African (~500 Ma).

7.2. Early Paleozoic metamorphic *P-T* path and tectonic implications

A few studies have focused on the age of the Xinghuadukou Complex (Ge et al., 2015; Miao et al., 2007; Shanghu and Zheng, 2012; Wu et al., 2012). Mineral assemblages of various rocks from the Xinghuadukou Complex are described by Biao et al. (1999) and HBGMR (1993). Although research on preliminary ages and geochemistry data has been reported, a reliable metamorphic condition and reliable *P-T* for the Xinghuadukou Complex has not been identified.

Based on our studies, the Xinghuadukou Complex is characterized by a clockwise *P-T* path that includes a near-isobaric cooling part (Fig.). This clockwise *P-T* path can be subdivided into three main stages: a prograde stage, a near-peak upper amphibolite facies stage, and a near-isobaric cooling stage. The three stages of this clockwise *P-T* path provide a significant insight into the post-collision orogenic process and allow one to establish a model for the tectonic evolution of the Xinghuadukou Complex. The prograde metamorphic stage corresponds to the garnet growth and occurred as prograde metamorphism at *P-T* conditions of 640 and 6.1 kb (point A). The metapelites from the Xinghuadukou Complex document the collision process between the Erguna Massif and the Xing'an Massif, and they underwent amphibolite facies metamorphism. During the collision and an increase in temperature, pressure, and depth, the metapelites experienced fluid-saturated solidus melting, muscovite dehydration melting, and part-of-biotite dehydration melting reactions to reach near-peak upper-amphibolite facies metamorphism with *P-T* conditions of 711–722 °C and 5–6 kb. Thereafter, during a near-isobaric cooling, the rock experienced retrograde metamorphism with muscovite crystallization forming in the core of biotite and sillimanite to reach the observed mineral assemblage at *P-T* conditions of 625 °C and 4.4 kb.

The timing of collision between the Erguna and the Xing'an massifs has been studied by many researchers for several decades. The most commonly quoted age of collision is from ~647 Ma until ~500 Ma. The Xinlin-Gaxian-Jifeng ophiolitic melanges crop out in the northern Great Xing'an Range, which marks the closure of the Xinlin-Xiguitu Ocean between the Erguna and Xing'an massifs, having U-Pb ages of 539–510 Ma (Xinlin), ~630 Ma (Gaxian), and 647 Ma (Jifeng) (Feng et al., 2016; Feng, 2015). The presence of the Toudaoqiao blueschist of N- normal-mid-ocean ridge basalt (MORB) and ocean island basalt (OIB) protoliths with an age of ~ 500 Ma along the south margin indicates the final collision between the Erguna and the Xing'an massifs (Miao et al., 2015;

Zhao et al., 2017; Zhou et al., 2015).

Widespread Late Paleozoic granites and diorites formed along the Xinlin-Xiguitu Suture including the Luguhe granite with an age of ~510 Ma (Wu et al., 2005b), the Tahe intrusion with an age of ~500 Ma (Ge et al., 2005), the Menduli granite with an age of ~502 Ma (Qin et al., 2007), the Halabaqi granite with an age of ~500 Ma (Sui et al., 2006), the Xikouzi granite with an age of ~500 Ma (Sun et al., 2013a), and the Hanjiayuanzi diorite with an age of 501 Ma (Zhao, 2017). The geochemical features of all these rocks indicate they formed in a post-collision or post-orogenic tectonic setting generated by the collision of the Erguna and the Xing'an massifs.

8. Conclusions

A detailed and systematic petrological, zircon-U-Pb-age and metamorphic study of metapelites within the Xinghuadukou Complex has led to the following primary conclusions:

- (1) The protoliths of the Sil-Grt-Bt-Ms paragneiss within the Xinghuadukou Complex from the Lulin Forest of NE China are claystone.
- (2) Garnet porphyroblasts in the metapelites show diffusion zoning from core to rim with inclusions of sillimanite, biotite and quartz. From the analysis of different mineral assemblage, the metamorphic evolution can be divided into three stages: an early prograde metamorphic stage, a near-peak upper amphibolite facies stage, and a near-isobaric cooling stage.
- (3) Zircon U-Pb dating yielded metamorphic ages between 496 and 509 Ma and an upper limit deposition age of at least ~601 Ma, which indicates the Xinghuadukou Complex formed during the Neoproterozoic, rather than during the Paleoproterozoic to Mesoproterozoic, and recorded Pan-African metamorphic events of the Erguna Massif.
- (4) The *P-T* conditions together with mineral assemblages define a clockwise *P-T* path, revealing important information related to the post-collision tectonic setting of the Erguna and Xing'an massifs.

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Fig.1.

Fig.2. Simplified geological map of the Lulin Forest area in northern Great Xing'an Range (modified from Heilongjiang Bureau of Geology and Mineral Resources(1993))

Fig.3.Field photographs of outcrop within the Xinghuadukou Complex in Lulin Forest from the Erguna Massif

Fig.4. Microphotographs of within the Xinghuadukou Complex in Lulin Forest from the Erguna Massif

Fig.5. Compositional zoning of garnet from Sample Z1545-9 from the Xinghuadukou Complex

Fig.7. Representative cathodoluminescence (CL) images of zircons from metapelite sample Z1545-5(a), metapelite sample Z1545-10 (b), respectively, showing U-Pb analytical spots and corresponding ages.

Fig.8. LA-MC-ICP-MS U-Pb concordia diagrams and ages histogram and relative probability plots for sample Z1545-5 and Z1545-10 from the metapelites of the Xinghuadukou Complex