In-situ trace element analyses and Pb-Pb dating of zircons in granulite from Huangtuling, Dabieshan by LAM-ICP-MS

WU Yuanbao (吴元保)[1], CHEN Daogong (陈道公)[1], XIA Qunke (夏群科)[1], TU Xianglin (涂湘林)[2], CHENG Hao (程　昊)[1] & YANG Xiaozhi (杨晓志)

1. Department of Earth and Space Sciences, University of Science and Technology of China, Hefei 230026, China;
2. Guangzhou Institute of Geochemistry, Chinese Academy of Sciences, Guangzhou 510640, China
Correspondence should be addressed to Wu Yuanbao (email: dgchen@ustc.edu.cn)

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Abstract It is revealed by CL images that there are multi-stage growth internal structures of zircons in the Huangtuling granulite, including the inherited zircons, protolith zircons, sector and planar zone zircons and retrograde zircons. *In-situ* trace element compositions and Pb-Pb ages have been analyzed by LAM-ICP-MS. The sector and the planar zone domains show typical trace element characteristics of granulite zircon (low Th, U, Th/U, total REEs, clear negative Eu anomalies, relatively depleted HREE and small differential degree between MREE and HREE, etc.), indicating that they formed during granulite-facies metamorphism. The protolith zircons have trace element characteristics of crustal zircon (high Th, U, Th/U, total REEs and enriched HREEs, etc.). 12 analyzed spots on granulite-facies domains give a weighted mean $^{207}Pb/^{206}Pb$ age of (2154±26) Ma (MSWD = 3.8), which is the best estimated age of granulite-facies metamorphism of this sample. The weighted mean $^{207}Pb/^{206}Pb$ age of 5 analyzed spots on protolith zircon domains is (2714 ± 22) Ma (MSWD = 1.4), which represents the protolith forming time. The discovery of ca. 3.4 Ga inherited zircon indicates that there are Palaeoarchean continental materials in this area. The interpretation of formation conditions and the ages of zircons can be constrained by simultaneous *in-situ* analysis of trace elements and ages.

Keywords: granulite, zircon, trace element, Pb-Pb age, LAM-ICP-MS.

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Zircon is an accessory mineral occurring in many types of rocks. For the rich content of U and low content of common Pb, it is the principal mineral used for U-Th-Pb dating. It can be survived during weathering, transiting, high-grade metamorphism and even high temperature melting, because of its stabilization. On the other hand, the diffusion closure temperature of U-Pb for zircon is ca. 900℃[1], which is the highest among those of the mineral isotope diffusion systems. So it can partly or entirely keep its original radiogenic Pb after experiencing multi-stage geological events. In complex evolution area, zircons always have multi-stage growth internal structures. They can record the ages of different geological events. But it is difficult to get precise ages of this kind of zircons by the traditional TIMS technique, and to relate the ages to geological events. Recently, sensitive high-resolution ion microprobe (SHRIMP)[2] and laser micro-probe inductively coupled plasma-mass spectrometry (LAM-ICP-MS)[3−5] can analyze *in situ* zircon ages, and have the ability to get the protolith, metamorphic overgowth and inherited source zircon' s ages. These are very important for zircons with complex structures. However, it is difficult to give the geological meaning of zircon ages, especially to relate the metamorphic zircon's ages with specific P-T conditions in complex metamorphic rocks. The trace element characteristics of zircons are related not only to the ability of each element's entry into the zircon's lattices, but also to the concurrent growth minerals (such as garnet, feldspar, etc.). The presence or not of these minerals can be indications of specific metamorphic conditions (i.e. eclogite, amphibolite, granulite facies)[6−8]. Moreover, the trace element analyses and U-Pb dating of zircon can be done in the same domain[3,4]. The CL images can reveal very clearly the internal structures of zircons. These can lay the analyzed spots in a single-growth domain. The zircons' U-Pb can be interpreted more reasonably based on these techniques.

There are a few felsic-intermediate granulite outcrops in the Dabieshan area (such as the Huilanshan and the Huangtuling granulites). It is very important to research this typical low-crustal rock, for it can give useful information about the tectonic evolution, crust-mantle interaction and tectonic affinity of the Dabieshan area[9−11]. However, there are no precise ages of the protolith and the granulite-facies metamorphism yet[11−14]. The present paper studies the internal structures of zircons in Huangtuling granulite by CL, and simultaneously analyzes Pb-Pb isotopic ages and trace elements in different domains of zircons using the LAM-ICP-MS technique. These precisely confine the protolith and granulite-facies metamorphic ages of this area, and constrain the evolution history of this kind of rock.

1 Samples and analytical methods

The Northern Dabieshan granulite sample (HTL) was collected from the Huangtuling Primary School, Luotian County, Hubei Province. It is a dark brown biotite-plagioclase granulite, which contains an assemblage of garnet (~10%), plagioclase (~35%), quartz (~30%), biotite (~15%), minor hornblende, cordierite and some accessory minerals. The granulite-facies minerals are garnet+plagioclase+quartz+biotite+rutile/ilmenite+magnetie+zircon, the retrograde metamorphic minerals are hornblende and biotite, and pryoxene+cordierite coronal around garnet. Based on detailed petrological studies, Chen et al. (1998)[15] estimated that the P-T conditions of the peak and retrograde metamorphisms are 13.2 ± 2 ($\times 10^8$ Pa), 850 ± 50℃, and 6 ($\times 10^8$ Pa), 700℃, respectively. A 2-kg portion of sample was processed by standard techniques (Wilfley table, methylene iodide, and Frantz magnetic separator). Zircons were prepared as mineral separates, mounted in epoxy and polished down to expose the grain centers. The trace elements and Pb-Pb isotopic ages were analyzed on the basis of the CL images, which were done at the Nancy1 University, France.

Zircon trace element and Pb-Pb isotopic age analyses were performed on the Perkin-Elmer Sciex ELAN6000 ICP-MS coupled to a CETAC LSX-100 Laser Probe at the Guangzhou Institute of Geochemistry, the Chinese Academy of Sciences. The analytical conditions of the LAM-ICP-MS are as follows: a frequency-quadrupled Nd-YAG UV laser system was operating at 266 nm; the ablation pits were (20—30) μm × (20—40) μm; the operating mode was Q-switched, with maximum energy of 3 mJ/pulse; the power of ICP was 1000 W; the mass resolution of the quadrupole mass analyzer was (0.7 ± 0.02) amu, AC rod offset voltage was 15.4 V; the produced aerosol was transported into the ICP-MS by an Ar gas stream; the mass discrimination factor was determined by analyzing glass NIST610 as an external standard, and the selected Si as an internal standard for the content of SiO_2 in zircon equals 32.9%. Precisions of most trace elements are typically 5%—15% (RSD), but up to 20%—40% for a few trace elements with concentrations close to the limits of detection. Isobaric interference of ^{204}Hg overlapping ^{204}Pb was automatically corrected by the ^{202}Hg. The fractionation between ^{206}Pb and ^{207}Pb is not adjusted, for the fractionation adjustment of them is not ensured, and the isotopic fractionation of the same element is small. The ^{206}Pb/^{204}Pb ratios of samples are supposed to be mostly > 1000, because the counts of ^{204}Pb during sample analyses were the same as that of the background (less than 10), and the counts of ^{206}Pb are more than 1×10^4. So the common Pb does not need correcting for determining the radiogenic ^{207}Pb/^{206}Pb ages[16,17]. The detailed analytical method has been reported by Li et al. (2000)[4] and Liang et al. (1999)[3].

2 Results and discussion

2.1 CL images

The typical CL images of Huangtuling zircons are shown in fig. 1. Round multi-facies zircons mainly consist of sector zones, and parts of them have cores (fig. 1(a)). Planar zones are the main domains of short prismatic zircons. This kind of zircons commonly has cores (fig. 1(b)). The sector and planar zone domains are typical metamorphic zircon's characteristics[18, 19]. Most of the cores have magmatic oscillatory zones (fig. 1(b) and (c)), and may be the protolith zircons. The relationships between the sector and the planar zones of most zircons are not clear, but it can be seen from fig. 1(b) that the planar zone domain grows later than that of the sector zone. The zircon of fig. 1(b) has double structure-inherited cores, and the inner core is probably an old inherited zircon of the proto-

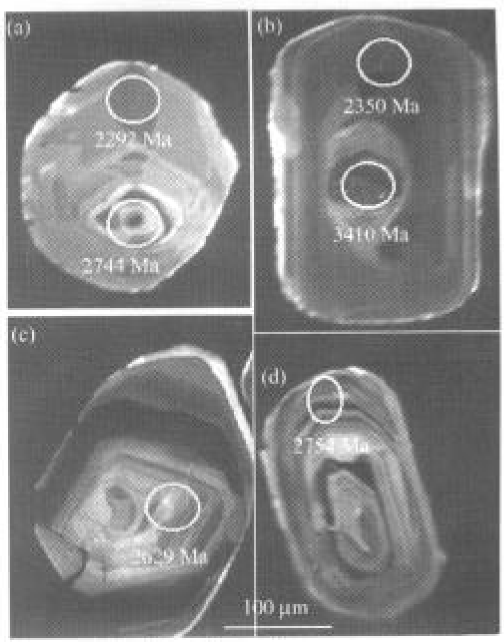

Fig. 1. Typical CL images of zircons in the Huangtuling granulite and spots locations. (a) Sector zone zircon. (b) and (c) Planar zone zircons. (d) Protolith zircon.

lith. There are bright white rims around the sector and the planar zone domains. They were probably formed during the retrograde metamorphism. Another kind of zircons are short prismatic with clear oscillatory zones (fig. 1(d)), they may be the protolith magmatic zircons.

2.2 Trace elements

The trace elements analyzed by LAM-ICP-MS are listed in table 1. The trace element characteristics are variable among different kinds of zircon domains.

The U contents of sector zone domains range from 179.0×10^{-6} to 321.8×10^{-6}, the Th contents range from 6.1×10^{-6} to 28.4×10^{-6}, and the Th/U ratios range from 0.03 to 0.13. Those of the planar zone domains are 171.6×10^{-6}—455.9×10^{-6}, 11.7×10^{-6}—22.0×10^{-6} and 0.04— 0.13, respectively. The zircons of low Th and U contents and Th/U ratios are typical metamorphic, which consist with their images. The U and Th contents (1231.7×10^{-6}—3660.2×10^{-6}, 74.6×10^{-6}—533.5×10^{-6}) and Th/U ratios (0.22—0.96) in the oscillatory domains are higher than those of the metamorphic zircons, and belong to the typical magmatic zircons.

The total REE contents and the chondrite-normalized REE patterns of zircons are quite different (fig. 2). The total REE contents of metamorphic domains (both sector zone and planar zone) are low, ranging from 99.6×10^{-6} to 318.3×10^{-6}. Those of the protolith zircons are higher, and more variable (1231.7×10^{-6}—3660.2×10^{-6}). Although there are some debates on whether the

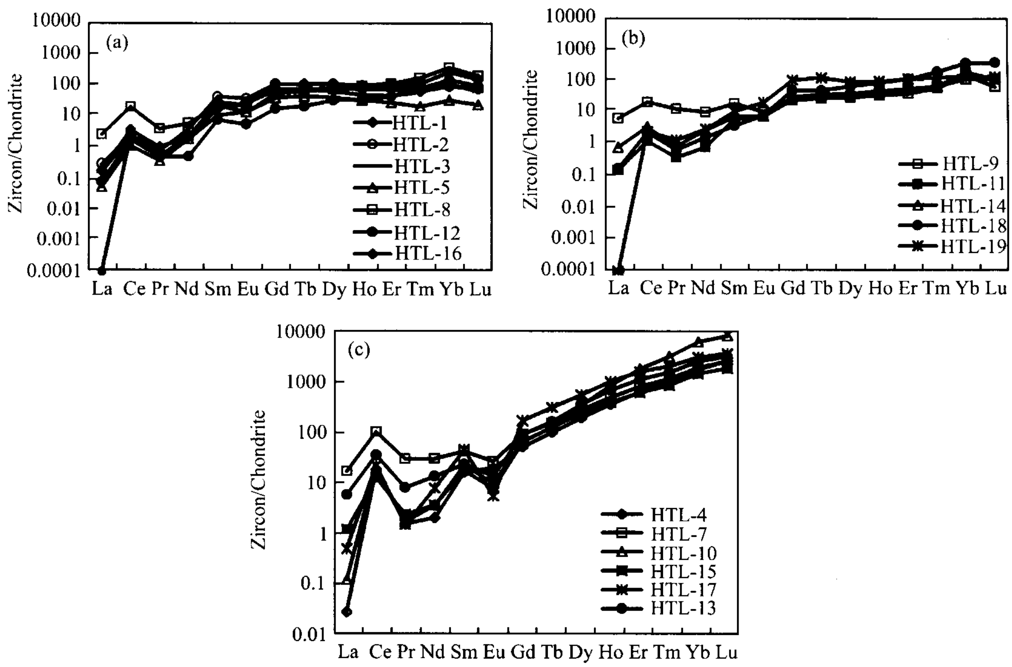

Fig. 2. Chondrite normalized REE patterns of different zircon domains in the Huangtuling granulite. (a) Sector zone domains. (b) Planar zone domains. (c) Protolith and inherited domains.

Table 1 Trace element results of zircons in Huangtuling granulite by LAM-ICP-MS ($\times 10^{-6}$)

Sample	Sector zone							Planar zone					Protolith					Inherited
	HTL-1	HTL-2	HTL-3	HTL-5	HTL-8	HTL-12	HTL-16	HTL-9	HTL-11	HTL-14	HTL-18	HTL-19	HTL-4	HTL-7	HTL-10	HTL-15	HTL-17	HTL-13
Th	9.7	13.4	8.2	6.1	28.4	8.6	16.8		11.7	17.9	17.8	14.8	120.2	331.2	533.5	74.6	149.7	315.3
U	211.3	233.6	204.5	183.0	222.8	179.0	321.8	171.6	214.8	437.5	455.9	249.1	185.6	343.6	739.3	334.4	208.2	679.1
La		0.07	0.04	0.01	0.62	0.02	0.04	1.54	0.04	0.19	0.04		0.01	4.16	0.03	0.30	0.12	1.42
Ce	1.6	1.5	1.4	0.7	12.6	1.1	2.3	13.1	0.8	2.2	1.5	1.6	11.9	64.1	12.4	8.2	14.2	22.6
Pr	0.0	0.1	0.0	0.0	0.4	0.0	0.1	1.2	0.0	0.1	0.1	0.1	0.2	2.8	0.2	0.2	0.1	0.8
Nd	1.1	1.8	0.8	0.9	2.7	0.2	1.0	4.7	0.4	1.3	0.8	1.4	1.0	14.3	1.7	1.7	3.8	6.5
Sm	2.7	6.3	2.8	1.6	3.4	1.1	4.3	2.8	0.9	1.1	0.6	1.7	2.5	6.4	2.7	3.5	6.9	3.6
Eu	1.4	2.1	1.5	0.8	0.8	0.3	1.3	0.6	0.4	0.4	0.5	1.2	1.1	1.5	0.5	0.6	0.3	0.8
Gd	15.5	22.3	13.4	7.7	10.6	3.5	21.0	4.8	4.9	6.1	9.9	20.5	10.6	18.7	12.9	13.5	34.3	18.6
Tb	2.6	3.8	2.5	1.6	2.3	0.8	4.0	1.0	1.1	1.3	1.8	4.4	3.8	5.7	4.8	4.6	11.5	6.1
Dy	19.7	27.6	15.5	9.9	17.9	8.1	27.5	7.2	8.2	9.5	16.3	22.5	48.5	70.3	86.9	60.6	138.1	90.9
Ho	4.0	5.2	2.7	1.8	5.2	1.9	5.4	1.9	2.3	2.5	4.6	5.3	20.2	27.2	46.3	21.7	56.1	38.3
Er	11.7	15.7	7.3	4.6	17.9	7.1	18.7	6.2	8.6	8.8	18.1	18.1	102.9	131.7	290.1	100.5	255.3	181.4
Tm	1.6	2.1	1.5	0.5	4.3	1.9	2.8	1.4	1.7	1.6	4.8	3.1	25.1	28.8	79.2	21.3	50.8	38.1
Yb	14.7	14.8	17.4	5.3	59.1	23.7	42.1	24.0	28.8	18.7	57.6	23.0	286.5	309.2	996.4	235.0	490.8	414.7
Lu	1.9	2.1	1.5	0.6	5.0	2.3	3.9	1.6	2.8	2.8	9.3	3.2	64.2	64.0	201.9	45.5	89.9	79.6
Y	135.2	171.5	98.7	63.6	145.3	63.1	183.7	54.3	83.2	76.5	148.1	158.2	724.1	891.8	1924.2	714.6	1742.5	1233.6
REE+Y	214.0	277.1	167.2	99.6	287.9	115.4	318.3	126.6	144.2	133.2	273.9	264.0	1303.0	1641.0	3660.2	1231.7	2894.8	2137.2
Ce/Ce*	9.8	4.6	7.4	5.5	6.2	6.3	6.2	2.2	4.8	4.3	5.8	3.7	23.4	4.3	21.2	7.1	22.3	7.08
Eu/Eu*	0.53	0.48	0.62	0.56	0.35	0.44	0.34	0.49	0.48	0.4	0.3	0.36	0.57	0.4	0.19	0.23	0.05	0.23
Th/U	0.05	0.06	0.04	0.03	0.13	0.05	0.05	0.13	0.05	0.04	0.04	0.06	0.65	0.96	0.72	0.22	0.72	0.46

REE contents of zircons can imply the type of their source rocks, it is well received that the total REE contents of zircons from mantle-derived rocks (e.g. kimberlite, syenite, carbonatite, etc.) are low ($<300\times10^{-6}$)[4,20]. It is suggested that the protolith of the Huangtuling granulite be crustal rock by its high total REE contents.

All the analyzed domains have clear positive Ce and negative Eu anomalies (fig. 2). The metamorphic zircon has less pronounced Ce and Eu anomalies (Ce/Ce* = 4.6—9.8, Eu/Eu* = 0.34—0.62 for sector zone zircons; Ce/Ce*=2.2—5.8, Eu/Eu* = 0.36—0.49 for planar zone zircons, respectively). The protolith zircons have more significant and variable positive Ce and negative Eu anomalies (Ce/Ce* = 4.3—23.4, Eu/Eu* = 0.05—0.57), similar to those of the crustal zircons[4]. A positive Ce anomaly is connected with the presence of Ce^{4+}, which is prior to substituting into the Zr site. The metamorphic zircons of negative Eu anomalies are considered to be equilibrate with the feldspars, for the Eu^{2+} ions are preferential entry into them[6]. It is indicated that the sector and the planar zone zircons grow at the presence of feldspar by the clear negative Eu anomalies of them.

All zircons are LREE-depleted, HREE-enriched in the chondrite-normalized REE patterns (fig. 2). The enrichments of HREE are different among zircons. The depletion of LREE in zircons can be quantified by the chondrite-normalized $(Nd/Yb)_{CN}$ values, because the contents of La and Pr are very low and Ce shows an anomaly[4]. The protolith zircons show strong enrichment of HREE with $(Nd/Yb)_{CN}$=0.0006—0.0161, and high contents of LREE (fig. 2), which are typical REE characteristics of zircons from crustal rocks; whilst the metamorphic zircon domains show weak enriching of HREE [$(Nd/Yb)_{CN}$=0.0035—0.056 for sector zone zircons, and $(Nd/Yb)_{CN}$=0.0045—0.069 for planar zone zircons], low LREE contents, small differential degree between MREE and HREE, and horizontal patterns in fig. 2(b) and 2(c). These features indicate that the metamorphic zircons are equilibrated with garnets, which strongly enrich HREE[6−8].

The REE characteristics of inherited zircon in protolith are similar with the protolith zircons, suggesting that it is also magmatic zircon.

From the discussion above, we can see that the metamorphic zircons (both sectorial zone and planar zone ones) equilibrate with garnet and feldspar. It is argued that the metamorphic zircons are formed during the granulite-facies metamorphism, for the coexistence of garnet and feldspar denote granulite-facies conditions. The granulite-facies metamorphic event can be precisely dated by these domains[8]. The protolith age can be obtained by dating the protolith magmatic domains.

2.3 Pb-Pb ages

Pb isotopic data and ages are present in table 2. The $^{207}Pb/^{206}Pb$ ages of sectorial zone domains range from 1928 Ma to 2339 Ma, those of the planar zones range from 2049 Ma to 2350 Ma. Although the sectorial zones grow earlier than the planar zones, they are in good agreement within analytical errors. This phenomenon might be explained as below: (i) their ages are different, but they are concealed by the analytical errors of LAM-ICP-MS; (ii) the age difference between them is small, for the granulite-facies event is short. The latter is confirmed by the garnet composition study of Chen et al. (1998)[15]. 12 spot analyses in metamorphic domains give an average $^{207}Pb/^{206}Pb$ age of (2154 ± 26) Ma (MSWD=3.8) (fig. 3), which is the precise time of the granulite metamorphism.

Table 2 Pb-Pb isotopic data of zircons in Huangtuling granulite

Sample	Isotopic ratio $^{207}Pb/^{206}Pb$	2*s*	$^{207}Pb/^{206}Pb$ age/Ma	±
HTL-1	0.1191	0.00774	1942	126
HTL-2	0.1182	0.01158	1928	189
HTL-3	0.1340	0.01085	2150	174
HTL-5	0.1288	0.01443	2081	233
HTL-8	0.1495	0.00942	2339	147
HTL-12	0.1410	0.00409	2239	65
HTL-16	0.1454	0.00960	2292	151
HTL-9	0.1398	0.03649	2224	580
HTL-11	0.1287	0.00836	2079	135
HTL-14	0.1504	0.01068	2350	167
HTL-18	0.1340	0.00080	2151	13
HTL-19	0.1265	0.01948	2049	316
HTL-4	0.1756	0.01036	2611	154
HTL-7	0.1914	0.00861	2754	124
HTL-10	0.1850	0.00463	2698	67
HTL-15	0.1902	0.00095	2744	14
HTL-17	0.1774	0.01739	2629	258
HTL-13	0.2887	0.02569	3410	303

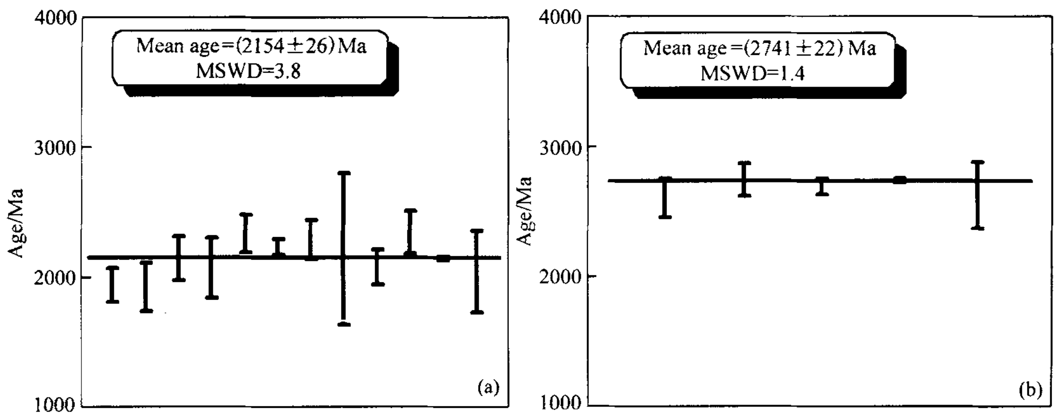

Fig. 3. LAM-ICP-MS zircon Pb-Pb ages for the Huangtuling granulite. (a) Granulite-facies metamorphic age. (b) Protolith age.

Five spots in protolith domains give an average $^{207}Pb/^{206}Pb$ age of (2741 ± 22) Ma (MSWD = 1.4) (fig. 3), which is the protolith age of this sample. The inherited zircon of protolith gives an oldest $^{207}Pb/^{206}Pb$ age of (3410 ± 303) Ma.

Chen et al. (1996)[12] firstly dated this granulite lens by zircon U-Pb TIMS, and got a discordant line with upper and lower intercept ages of (2663 ± 56) Ma and (1690 ± 82) Ma, which were interpreted as protolith or regional metamorphic and granulite-facies metamorphic ages, respectively. According to our CL images, there are multi-stage growth zircons (including granulite, protolith, retrograde metamorphic and inherited zircons) in the granulite. Bulk analyses of these complex grains by traditional TIMS technique usually give geological meaningless "mixing" ages. Jian et al. (1999)[11] used the zircon evaporation technique to date this granulite, obtained $^{207}Pb/^{206}Pb$ ages ranging from 1992 Ma to 2814 Ma, and regarded the minimum as the granulite metamorphic age, the maximum age as the protolith age. By the same reason as above, the minimum value may be considered being younger than the true granulite metamorphic age, and the maximum age being probably older than the protolith age. Zhou et al. (1999) [13] gained an age of (1998 ± 35) Ma by stepwise dissolution Pb-Pb dating of garnet, which is probablly a little younger than the granulite metamorphic age, for the garnet in this area always shows retrograde compositional zoning. A less precise Sm-Nd isochron age ((2238 ± 300) Ma) of pyroxene + feldspar + whole rock was obtained by Ma et al. (2000)[14], but the age error is too large. Our granulite metamorphism and protolith ages come from the zircon domains that are proven to form during granulite metamorpism and protolith magmatism by CL images and trace element characteristics. These ages are more precise, and can link with geological event more reliably. We can thus give useful constraints on its formation conditions and geological meaning to their ages by simultaneous *in-situ* analyses of the trace elements and isotopic ages of zircon.

The inherited zircon of the protolith gives a $^{207}Pb/^{206}Pb$ age of (3410 ± 303) Ma, which is the minimum age of this domain. We obtained the similar age by SIMS (Chen Daogong, unpublished data). The depleted mantle mode Nd age is also very old (T_{DM} = 3.1 Ga) of this granulite[14]. All these suggest that the very old age of this inherited zircon is reasonable. It is confirmed that there are very old (ca. 3.4 Ga) crustal materials in the Dabieshan area by our zircon ages. The ca. 3280 Ma xenocryatal zircons have been found in the meta-sedimentry rock from the Kongling high-grade metamorphic terrain of Hubei Province, which locates on the northern margin of Yangtze Block[21]. Zhang et al. (1990)[22] has also found inherited Pb with age of (3300 ± 180) Ma in zircon of quartz-syenite from Dalongshan, eastern Dabieshan, Anqing City, Anhui Province. Compared with the above data, the Huangtuling granulite can be supposed to be the old crustal materials from Yangtze Block. The same conclusion has also been proposed by Zheng et al. (2001)[9] based on the research of oxygen isotopes. It is suspected that the Palaeoarchean basement of Yangtze Block was sizable, which was demolished by later geological events.

3 Conclusion

In-situ trace elements compositions and Pb-Pb ages of zircons in the Huangtuling granulite have been analyzed by LAM-ICP-MS based on the detailed CL images. It is revealed by CL image technique that the zircons have complex internal structures, which include protolith, sectorial and planar zones, retrograde and old inherited zircons. There are very consistent relationships between the Th and U contents and Th/U ratios and the CL images. It is indicated by the REEs in zircons that the sectorial and the planar zone domains were formed during the granulite-facies metamorphism, and the protolith zircons came from typical crustal rocks. The protolith was formed in late Archean ((2741 ± 22) Ma). The age of granulite-facies metamorphism is (2154 ± 26) Ma. The detection of ca. 3.4 Ga inherited zircon indicates that there are Palaeoarchean crustal materials in this area.

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