



SIMS zircon U–Pb dating of the Late Cretaceous dinosaur egg-bearing red deposits in the Tiantai Basin, southeastern China

Huaiyu He^{a,*}, Xiaolin Wang^{b,*}, Qiang Wang^b, Shunxing Jiang^{b,c}, Xin Cheng^{b,c}, Jialiang Zhang^{b,c}, Zhonghe Zhou^b, Zikui Zhao^b, Yangen Jiang^d, Fangming Yu^e, Chenglong Deng^f, Jinhui Yang^f, Rixiang Zhu^f

^a Key Laboratory of the Earth's Deep Interior, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, China

^b Key Laboratory of Evolutionary Systematics of Vertebrates, Institute of Vertebrate Paleontology and Paleoanthropology, Chinese Academy of Sciences, Beijing 100044, China

^c University of Chinese Academy of Sciences, Beijing 100049, China

^d Tiantai Bureau of Land and Resources of Zhejiang Province, Tiantai 317200, China

^e Zhejiang Hydrogeology and Engineering Geology Team, Ningbo 315012, China

^f State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, China

ARTICLE INFO

Article history:

Received 21 January 2012

Received in revised form 22 October 2012

Accepted 6 November 2012

Available online 22 November 2012

Keywords:

SIMS U–Pb zircon geochronology

Late Cretaceous

Dinosaur eggs

Tiantai basin

China

ABSTRACT

Dinosaur eggs or fragments are abundant and extensively distributed in China. They can be very informative in biostratigraphic division and correlation of continental strata where other fossils are relatively lacking. Despite remarkable discoveries of vertebrate fossils, particularly dinosaur eggs and skeletons from the middle and Late Cretaceous of both northern and southern China, there is hardly any direct evidence for the ages of the vertebrate-bearing terrestrial deposits. To constrain their depositional ages, here we have obtained SIMS U–Pb zircon ages from the tuffs interbedded with dinosaur egg-bearing sediments from the Laijia and Chichengshan formations of the terrestrial red deposits of the Late Cretaceous in the Tiantai Basin, Zhejiang Province, southeastern China. The SIMS zircon U–Pb ages from the Laijia and Chichengshan formations are about 96–99 Ma (Cenomanian) and 91–94 Ma (Turonian), respectively, providing direct time constraints on the vertebrate and dinosaur egg evolution in the Late Cretaceous as well as a basis for correlation with terrestrial Cretaceous deposits in other regions of southern and northern China.

© 2012 Elsevier Ltd. All rights reserved.

1. Introduction

The middle–Late Cretaceous is an important period in the Mesozoic for vertebrate evolution, particularly for understanding the impact of the greenhouse temperatures in the middle Cretaceous upon their taxonomic diversification, body size and diversity change. Middle and Late Cretaceous terrestrial deposits are well developed in both southern and northern China. Many important taxa of dinosaurs including both complete skeletons and eggs have been reported, some of which bear important paleogeographic and evolutionary significance (Zhao, 1994; Zhao et al., 2009; Li et al., 2009a,b; Xu et al., 2010; Wang et al., 2010b,c, 2012).

In the past years, we have made several excavations in a number of Cretaceous localities in the Tiantai Basin, eastern Zhejiang Province (Fig. 1a and b), one of the typical regions of well developed terrestrial Cretaceous deposits in China. Recently, different dinosaur eggs have been reported and biostratigraphic division and correlation of continental strata have been discussed based on

* Corresponding authors.

E-mail addresses: huaiyuhe@mail.iggcas.ac.cn (H. He), wangxiaolin@ivpp.ac.cn (X. Wang).

these eggs (Fang et al., 2000, 2003; Jin et al., 2007; Qian et al., 2008a,b; Wang et al., 2010b,c, 2011; Zhang, 2010). However, the lack of sufficient and reliable chronostratigraphic data for the Tiantai Group in the region that bears the dinosaur bones and eggs has become a growing predicament for precise correlation between vertebrates-bearing deposits in various basins in eastern Zhejiang as well as with Cretaceous deposits in other regions of China.

In this paper we report the SIMS U–Pb zircon dating results from tuffs interbedded in the fossil-bearing red deposits of the Laijia and Chichengshan formations in Tiantai County, Zhejiang, providing direct ages and a preliminary time frame for studying the vertebrate evolution in the region in the Late Cretaceous (approximately from 99 Ma to 91 Ma).

2. Geologic setting, dinosaur egg fauna and sampling

Zhejiang Province is located in southeast China (Fig. 1a). The region is well known for developing both the Early Cretaceous terrestrial deposits with abundant fossil fishes (Chang and Chow, 1977) and Late Cretaceous deposits that contain dinosaur bones and eggs. They are distributed in many pull-apart basins filled with fluvial

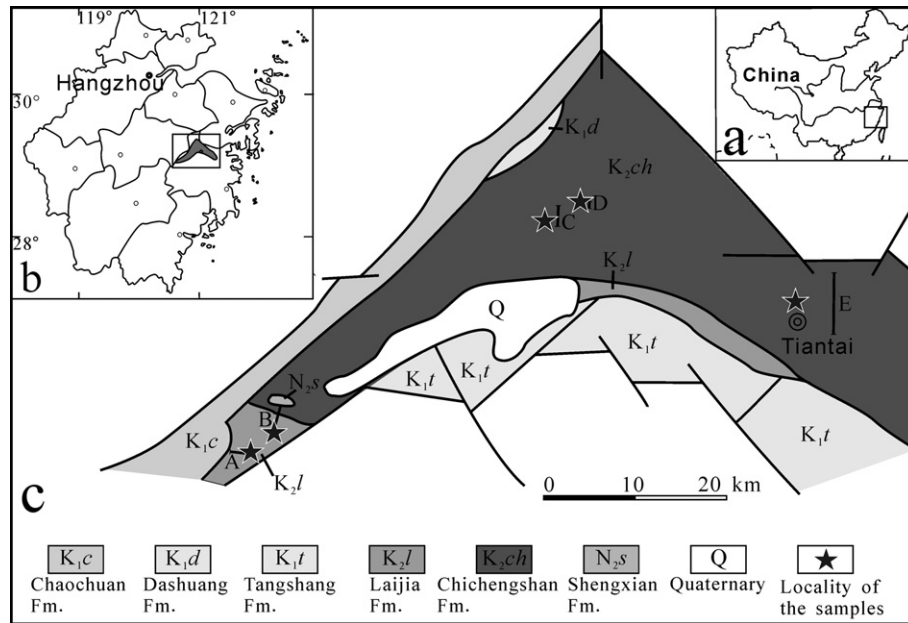


Fig. 1. (a) Sketch map showing the location of Zhejiang province. (b) Sketch map showing the location of Tiantai basin. (c) Mesozoic strata of the Tiantai basin and location of the studied sections (A–E). Modified from Wang et al. (2010c).

and lake sediments commonly with tuffs interbedded in some sections.

The late Mesozoic volcano-sedimentary rock series in eastern Zhejiang consists of the upper and the lower rock series, the former including the Upper Cretaceous Yongkang Group (Yongkang Basin, western Zhejiang), Qujiang Group (Quzhou-Jinhua areas, western Zhejiang) and Tiantai Group (Tiantai Basin, eastern Zhejiang), and the latter including the Lower Cretaceous Jiande Group (Jiande Basin, western Zhejiang) and Moshishan Group (Tiantai Basin, eastern Zhejiang). Previous studies, using K–Ar, Rb–Sr, U–Pb and Ar–Ar methods, of the volcanic rocks combined with biostratigraphic analysis have provided rough age estimates for those groups, e.g., 113–90 Ma for the Yongkang Group (see Luo and Yu, 2004 and references therein), 105–81 Ma for the Qujiang Group (see Yu et al., 2012 and references therein), and 109–94 Ma for the Tiantai Group (see Yu et al., 2010 and references therein). However, analytical procedures and detailed data are absent in the studies. It's thus hard to evaluate the quality of the dates. Recently, Wang et al. (2010a) have dated the feldspars from sediments of the Lower Cretaceous Moshishan Group in eastern Zhejiang Province by Ar–Ar method (single grain laser fusion) and estimated their temporal range to be 118–109 Ma.

The middle Cretaceous deposits in the Tiantai Basin (Fig. 1b and c) in eastern Zhejiang comprise the Tangshang Formation, Laijia Formation and Chichengshan formation from the bottom upwards (Fig. 2). The Laijia Formation comprises red fine sandstones and conglomerates. Most of the dinosaur eggs and bones have been found in the Laijia Formation and the bottom of the Chichengshan formation (Wang, 2010; Wang et al., 2011, 2012). Wang et al. (2010b,c) have recognized at least six layers of tuffs (from ten centimeters to more than 10 m thick, Fig. 2) interbedded within the sandstones. The Chichengshan formation also contains two layers of tuffs (Fig. 2). These tuff layers are conformably interbedded in the sandstones; both the lower and upper contacts of the tuff layers are sharp and planar. Hence, the dating of the tuffs provide a depositional age for the sedimentary layers that bracket the layer.

The dinosaur egg fauna of the Tiantai Basin represents one of the most important oofaunas in China with great diversity and abundance. It currently comprises seven oofamilies, 12 oogenera

and 15 oospecies (Wang, 2010; Wang et al., 2010b,c, 2011, 2012; Zhang, 2010). Among them, faveoololithids and dictyoolithids are most diverse and abundant, including three oofamily, five oogenus and seven oospecies, i.e., Faveoololithidae (*Parafaveoololithus*, *Hemifaveoololithus*), Similifaveoololithidae (*Similifaveoololithus*) and Dictyoolithidae (*Dictyoolithus*, *Paradictyoolithus*). The Macroelongatoolithidae is found in the Tiantai Basin of Zhejiang Province and the Xixia Basin of Henan Province in central China, including two oogenus and two oospecies in the Tiantai Basin: *Macroelongatoolithus* and *Megafusoolithus*. In addition, some newly known ootaxa *Paraelongatoolithus* (Elongatoolithidae), *Prismatoolithus* (Prismatoolithidae), *Stalicoolithus* and *Coralloidoolithus* (Stalicoolithidae), and *Mosaioolithus* have also been found in the Tiantai Basin.

The dating samples in this study come from three stratigraphic sections of two formations in the Tiantai Basin, Zhejiang: samples T071201-01, T071201-04 and T071201-05 from the Laijia Formation; and samples T071202-01, T071202-02 and T071203-01 from the Chichengshan formation (Fig. 2). Thin section studies show that there are no significant compositional difference between these samples, and they are composed of slightly altered feldspar (30–40%), quartz (30–40%), altered biotite (10–15%), rock fragments (5–10%) and accessory minerals such as zircon and opaque minerals.

3. Analytical methods

Thin section studies showed that the feldspars in the tuff samples were slightly altered, and not suitable for $^{40}\text{Ar}/^{39}\text{Ar}$ dating. Thus we separated zircons for SIMS U–Pb dating. The samples were processed by conventional magnetic and density techniques to concentrate non-magnetic, heavy fractions. Zircon grains, together with zircon standard TEMORA 2 were mounted in epoxy which was then polished to section the crystals for analysis. All zircons were documented with transmitted and reflected light micrographs as well as cathodoluminescence (CL) images to reveal their internal structures, and the mount was vacuum-coated with high-purity gold.

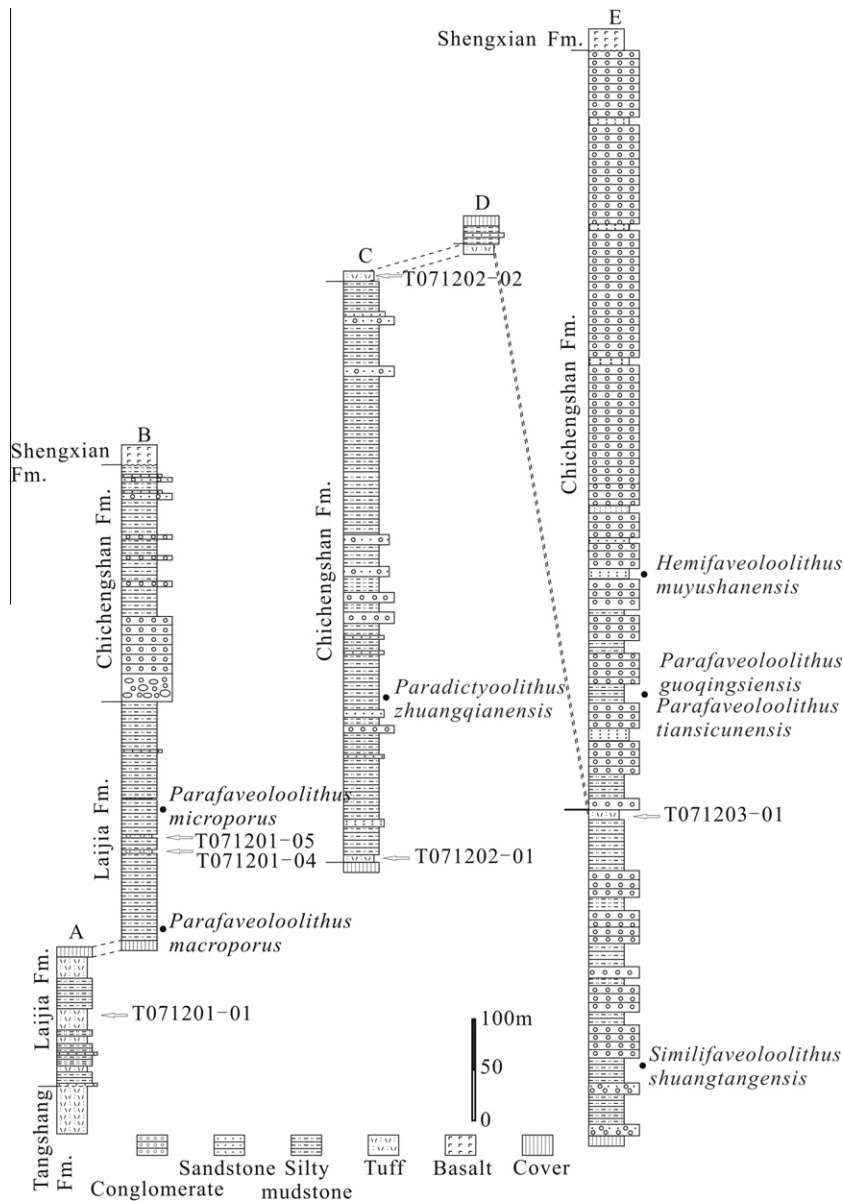


Fig. 2. Correlation and stratigraphy of the sections (A–E) in Fig. 1. The arrows indicate the position of the samples. Tuff T071202-02 in section C and tuff T071203-01 in section E are supposed to be the same layer. Modified from Wang et al. (2012).

Measurements of U, Th and Pb were conducted using the Cameca IMS-1280 ion microprobe at the Institute of Geology and Geophysics, Chinese Academy of Sciences in Beijing. Operating and data process procedures were similar to those described by Li et al. (2009a,b); U–Th–Pb ratios were determined relative to the standard zircon TEMORA 2 ($^{206}\text{Pb}/^{238}\text{U}$ age of 416.5 ± 0.2 Ma) (Black et al., 2003), analyses of which were interspersed with known grains. Because the standard zircon 91500 is the best currently available for a variety of element abundances and isotopic compositions (Ireland and Williams, 2003), absolute abundances of U/Th were determined relative to 91500 (Wiedenbeck et al., 1995) on a separate mount. The mass resolution used to measure Pb/Pb and Pb/U isotopic ratios was 5400 during the analyses. Measured compositions were corrected for common Pb using non-radiogenic ^{204}Pb . Corrections are sufficiently small to be unaffected by the choice of common Pb composition; an average of present-day crustal composition (Stacey and Kramers, 1975) was used for the common Pb assuming that the common Pb is largely due to surface contamination introduced during sample preparation. Be-

cause the O_2 primary ion beam was accelerated at 13 kV, with an intensity of ca. 10 nA, combined oxygen flooding technique which can significantly enhance secondary Pb^+ ion, our signal is improved about four times (about 21 cps/ppm/nA) and uncertainty of U/Pb calibration is less than 1.5% (Li et al., 2010a,b). SIMS U–Pb zircon data are presented in Table 1. Uncertainties for each individual analysis are reported at a 1σ level; mean ages for pooled U/Pb and Pb/Pb analyses are quoted with 95% confidence intervals. Decay constants are those recommended by the IUGS Subcommittee on Geochronology (Steiger and Jaeger, 1977). The uncertainty of standard (0.05%) and decay constants (0.1%) was propagated into the final ages.

4. Results

Zircons from the six samples are mostly euhedral, transparent and colorless, and have length to width ratios between 1:1 and 5:1. Most grains have oscillatory zoning in CL images (Fig. 3), while

Table 1
SIMS zircon U–Pb data.

Sample/ Spot #	[U] ppm	[Th] ppm	[Pb] ppm	Th/U meas	f ₂₀₆ %	²⁰⁷ Pb/ ²³⁵ U	±σ %	²⁰⁶ Pb/ ²³⁸ U	±σ %	ρ	²⁰⁷ Pb/ ²³⁵ U	±σ	²⁰⁶ Pb/ ²³⁸ U	±σ
T07120101-01@1	51	86	1	1.69	8.67	0.09936	5.55	0.0148	1.92	0.35	96.2	5.1	94.5	1.8
T07120101-01@2	125	174	3	1.40	3.87	0.10092	3.60	0.0156	1.72	0.48	97.6	3.4	99.6	1.7
T07120101-01@3	19	37	0	1.97	10.48	0.09299	11.96	0.0143	2.14	0.18	90.3	10.4	91.4	1.9
T07120101-01@4	15	27	0	1.79	3.23	0.11156	10.98	0.0165	2.19	0.20	107.4	11.2	105.2	2.3
T07120101-01@5	18	32	0	1.80	9.83	0.10522	11.21	0.0158	2.23	0.20	101.6	10.9	101.3	2.2
T07120101-01@6	15	18	0	1.16	2.21	0.09938	17.22	0.0155	3.38	0.20	96.2	15.9	99.3	3.3
T07120101-01@7	46	93	1	2.00	7.76	0.10035	6.57	0.0153	1.85	0.28	97.1	6.1	97.6	1.8
T07120101-01@8	18	35	0	1.92	6.81	0.10932	8.93	0.0162	2.50	0.28	105.3	9.0	103.9	2.6
T07120101-01@9	16	30	0	1.91	5.81	0.10352	13.80	0.0156	3.09	0.22	100.0	13.2	99.8	3.1
T07120101-01@10	14	27	0	1.92	6.88	0.10326	13.13	0.0157	2.20	0.17	99.8	12.6	100.6	2.2
T07120101-01@11	16	27	0	1.72	11.33	0.10094	13.15	0.0149	1.96	0.15	97.6	12.3	95.4	1.9
T07120101-01@12	17	34	0	1.92	7.78	0.10103	10.41	0.0154	2.02	0.20	97.7	9.7	98.5	2.0
T07120101-01@13	16	20	0	1.24	4.80	0.10728	9.75	0.0159	3.05	0.31	103.5	9.6	101.4	3.1
T07120101-01@14	74	96	2	1.30	3.14	0.10260	4.54	0.0161	1.86	0.41	99.2	4.3	103.0	1.9
T07120101-01@15	79	95	2	1.20	2.92	0.11304	4.15	0.0163	1.77	0.43	108.7	4.3	104.1	1.8
T07120101-01@16	26	50	1	1.97	7.21	0.10264	7.77	0.0152	2.03	0.26	99.2	7.4	97.3	2.0
T07120101-01@17	23	46	1	2.01	6.19	0.08976	8.89	0.0153	2.69	0.30	87.3	7.5	98.1	2.6
T07120101-01@18	20	40	1	1.99	9.00	0.10690	10.48	0.0156	2.11	0.20	103.1	10.3	99.8	2.1
T07120101-01@19	23	46	1	1.98	4.47	0.10446	11.51	0.0150	2.17	0.19	100.9	11.1	96.3	2.1
T07120101-01@20	13	24	0	1.82	12.25	0.09795	9.94	0.0156	2.33	0.23	94.9	9.0	99.8	2.3
T07120101-01@21	25	50	1	1.96	7.49	0.09780	10.22	0.0149	2.06	0.20	94.7	9.3	95.5	1.9
T07120101-01@22	11	15	0	1.34	5.23	0.10688	11.74	0.0161	2.38	0.20	103.1	11.6	103.0	2.4
T07120101-01@23	17	34	0	1.94	9.54	0.10371	11.02	0.0162	2.16	0.20	100.2	10.6	103.5	2.2
T07120101-01@24	31	58	1	1.86	7.59	0.09782	8.83	0.0147	1.86	0.21	94.8	8.0	94.0	1.7
T07120101-01@25	10	16	0	1.65	0.13	0.10306	10.99	0.0165	2.52	0.23	99.6	10.5	105.7	2.6
T071201-04@1	255	168	5	0.66	0.36	0.10306	2.72	0.0153	1.72	0.63	99.6	2.6	97.9	1.7
T071201-04@2	228	290	5	1.27	0.33	0.10220	2.83	0.0150	1.74	0.61	98.8	2.7	96.3	1.7
T071201-04@3	373	273	7	0.73	0.17	0.10246	2.98	0.0152	1.74	0.58	99.0	2.8	97.2	1.7
T071201-04@4	1572	1767	33	1.12	1.88	0.09585	3.58	0.0151	1.73	0.48	92.9	3.2	96.9	1.7
T071201-04@5	86	61	2	0.71	0.73	0.09232	6.75	0.0150	1.87	0.28	89.7	5.8	96.0	1.8
T071201-04@6	215	153	4	0.71	0.47	0.10347	3.30	0.0154	1.76	0.53	100.0	3.1	98.8	1.7
T071201-04@7	169	207	4	1.23	0.11	0.10452	3.61	0.0155	1.75	0.49	100.9	3.5	99.3	1.7
T071201-04@8	188	310	4	1.64	0.38	0.09790	3.06	0.0153	1.74	0.57	94.8	2.8	98.1	1.7
T071201-04@9	358	252	7	0.70	0.27	0.10470	2.80	0.0157	1.73	0.62	101.1	2.7	100.7	1.7
T071201-04@10	152	116	3	0.76	0.27	0.10163	3.16	0.0157	1.78	0.56	98.3	3.0	100.4	1.8
T071201-04@11	283	307	6	1.08	0.33	0.10267	2.64	0.0152	1.74	0.66	99.2	2.5	97.4	1.7
T071201-04@12	51	57	1	1.12	0.16	0.09071	5.25	0.0155	2.02	0.38	88.2	4.4	98.9	2.0
T071201-04@13	75	112	2	1.50	1.24	0.08384	8.97	0.0150	1.81	0.20	81.8	7.1	95.8	1.7
T071201-04@14	220	453	6	2.06	0.35	0.10467	2.83	0.0158	1.74	0.62	101.1	2.7	101.1	1.7
T071201-04@15	208	229	4	1.10	0.17	0.09929	3.66	0.0150	1.75	0.48	96.1	3.4	95.7	1.7
T071201-04@16	107	113	2	1.06	0.29	0.09791	4.49	0.0149	1.82	0.40	94.8	4.1	95.3	1.7
T071201-04@17	286	393	7	1.38	0.21	0.10083	2.93	0.0155	1.77	0.60	97.5	2.7	99.1	1.7
T071201-04@18	52	68	1	1.30	0.82	0.10098	4.88	0.0150	1.87	0.38	97.7	4.6	96.0	1.8
T071201-04@19	62	90	1	1.45	0.66	0.09773	7.74	0.0152	1.73	0.22	94.7	7.0	97.0	1.7
T071201-04@20	111	156	2	1.41	0.75	0.09727	8.18	0.0147	1.77	0.22	94.3	7.4	93.9	1.7
T071201-04@21	112	162	3	1.44	0.47	0.09954	4.47	0.0150	1.81	0.40	96.3	4.1	96.0	1.7
T071201-04@22	173	284	4	1.65	0.52	0.08859	6.34	0.0148	1.76	0.28	86.2	5.3	94.8	1.7
T071201-04@23	177	190	4	1.08	0.81	0.09298	4.73	0.0149	1.77	0.38	90.3	4.1	95.0	1.7
T071201-04@24	126	250	3	1.99	0.74	0.09968	3.43	0.0154	1.77	0.52	96.5	3.2	98.3	1.7
T071201-04@25	82	130	2	1.58	0.29	0.09884	4.80	0.0150	1.86	0.39	95.7	4.4	95.8	1.8
T071201-05@1	207	108	4	0.53	0.54	0.09849	2.90	0.0151	1.73	0.60	95.4	2.6	96.5	1.7
T071201-05@2	69	59	1	0.85	0.97	0.09243	4.50	0.0153	1.90	0.42	89.8	3.9	97.6	1.8
T071201-05@3	60	83	1	1.39	0.74	0.09569	7.87	0.0143	1.77	0.23	92.8	7.0	91.6	1.6
T071201-05@4	80	75	2	0.93	0.86	0.09779	7.06	0.0149	1.79	0.25	94.7	6.4	95.3	1.7
T071201-05@5	227	156	4	0.69	0.22	0.10299	3.03	0.0151	1.78	0.59	99.5	2.9	96.7	1.7
T071201-05@6	380	247	7	0.65	0.18	0.09576	2.67	0.0151	1.73	0.64	92.9	2.4	96.4	1.7
T071201-05@7	269	280	6	1.04	0.12	0.10376	2.63	0.0152	1.75	0.66	100.2	2.5	97.0	1.7
T071201-05@8	116	144	2	1.24	0.64	0.08940	6.51	0.0139	1.97	0.30	86.9	5.4	89.3	1.7
T071201-05@9	18	13	0	0.73	2.34	0.10096	8.95	0.0150	2.13	0.24	97.7	8.4	95.7	2.0
T071201-05@10	27	30	1	1.11	1.96	0.09925	7.77	0.0153	2.01	0.26	96.1	7.2	97.9	1.9
T071201-05@11	310	291	6	0.94	0.24	0.09573	2.89	0.0152	1.74	0.60	92.8	2.6	96.9	1.7
T071201-05@12	120	153	3	1.27	1.03	0.10662	3.42	0.0153	1.74	0.51	102.9	3.3	98.2	1.7
T071201-05@13	144	182	3	1.27	0.71	0.08915	5.08	0.0144	1.76	0.35	86.7	4.2	92.4	1.6
T071201-05@14	90	140	2	1.55	0.86	0.09274	6.64	0.0153	2.11	0.32	90.0	5.7	97.7	2.0
T071201-05@15	162	117	3	0.72	0.14	0.10219	3.07	0.0154	1.73	0.56	98.8	2.9	98.3	1.7
T071201-05@16	39	49	1	1.24	1.78	0.10025	7.46	0.0154	1.99	0.27	97.0	6.9	98.7	1.9
T071201-05@17	89	131	2	1.47	0.67	0.09604	8.23	0.0151	1.88	0.23	93.1	7.4	96.7	1.8
T071201-05@18	179	302	4	1.69	0.54	0.09291	4.20	0.0150	1.77	0.42	90.2	3.6	95.8	1.7
T071201-05@19	72	107	2	1.49	0.71	0.09871	5.93	0.0149	1.84	0.31	95.6	5.4	95.3	1.7
T071201-05@20	350	201	7	0.57	0.12	0.10476	2.45	0.0159	1.73	0.70	101.2	2.4	101.7	1.7
T071201-05@21	187	302	4	1.61	0.69	0.09875	3.07	0.0146	1.96	0.64	95.6	2.8	93.6	1.8
T071201-05@22	43	39	1	0.91	0.65	0.10338	4.98	0.0157	1.81	0.36	99.9	4.8	100.5	1.8
T071201-05@23	128	197	3	1.54	0.48	0.09863	5.42	0.0148	1.76	0.33	95.5	4.9	94.9	1.7

(continued on next page)

Table 1 (continued)

Sample/ Spot #	[U] ppm	[Th] ppm	[Pb] ppm	Th/U meas	f_{206} %	^{207}Pb / ^{235}U	$\pm\sigma$ %	^{206}Pb / ^{238}U	$\pm\sigma$ %	ρ	^{207}Pb / ^{235}U	$\pm\sigma$	^{206}Pb / ^{238}U	$\pm\sigma$
T071201-05@24	130	119	3	0.91	0.46	0.09568	5.30	0.0151	1.83	0.35	92.8	4.7	96.6	1.8
T071201-05@25	362	235	7	0.65	0.29	0.09437	3.56	0.0146	2.08	0.58	91.6	3.1	93.3	1.9
T071201-05@26	273	270	6	0.99	0.15	0.09807	2.64	0.0152	1.74	0.66	95.0	2.4	97.4	1.7
T071201-05@27	22	17	0	0.79	1.82	0.09913	15.53	0.0148	2.19	0.14	96.0	14.3	94.7	2.1
T071201-05@28	83	135	2	1.63	2.19	0.10046	4.37	0.0150	1.81	0.41	97.2	4.1	96.1	1.7
T071201-05@29	71	47	1	0.67	1.11	0.10652	4.40	0.0154	1.76	0.40	102.8	4.3	98.7	1.7
T071202-01@1	153	125	3	0.81	0.64	0.09766	4.01	0.0147	1.70	0.42	94.6	3.6	93.9	1.6
T071202-01@2	141	178	3	1.27	2.07	0.09234	5.21	0.0145	1.64	0.31	89.7	4.5	93.0	1.5
T071202-01@3	172	203	3	1.18	1.31	0.09279	4.47	0.0143	1.60	0.36	90.1	3.9	91.6	1.5
T071202-01@4	91	79	2	0.87	1.54	0.09920	5.79	0.0149	1.67	0.29	96.0	5.3	95.5	1.6
T071202-01@5	193	161	4	0.84	1.16	0.09820	3.72	0.0146	1.57	0.42	95.1	3.4	93.7	1.5
T071202-01@6	70	84	1	1.20	1.99	0.09987	7.77	0.0147	1.71	0.22	96.7	7.2	94.1	1.6
T071202-01@7	349	295	7	0.85	0.68	0.09721	2.87	0.0149	1.54	0.54	94.2	2.6	95.2	1.5
T071202-01@8	379	282	7	0.74	0.78	0.10026	2.90	0.0146	1.52	0.52	97.0	2.7	93.3	1.4
T071202-01@9	95	69	2	0.73	1.29	0.10471	5.19	0.0152	1.65	0.32	101.1	5.0	97.6	1.6
T071202-01@10	142	107	3	0.75	0.80	0.09423	4.54	0.0147	1.60	0.35	91.4	4.0	94.4	1.5
T071202-01@11	173	241	4	1.40	1.50	0.08847	4.28	0.0145	1.58	0.37	86.1	3.5	92.9	1.5
T071202-01@12	139	169	3	1.22	0.20	0.10309	3.68	0.0149	1.57	0.43	99.6	3.5	95.1	1.5
T071202-01@13	115	118	2	1.03	1.78	0.09582	5.10	0.0151	1.64	0.32	92.9	4.5	96.9	1.6
T071202-01@14	150	161	3	1.07	1.45	0.09443	4.52	0.0147	1.62	0.36	91.6	4.0	93.8	1.5
T071202-01@15	150	200	3	1.34	1.26	0.10188	4.14	0.0152	1.58	0.38	98.5	3.9	97.4	1.5
T071202-01@16	152	124	3	0.82	1.65	0.08889	4.58	0.0146	1.57	0.34	86.5	3.8	93.6	1.5
T071202-01@17	105	96	2	0.92	1.90	0.09439	5.39	0.0142	2.13	0.39	91.6	4.7	91.1	1.9
T071202-01@18	78	140	2	1.79	2.79	0.09470	6.47	0.0145	1.65	0.26	91.9	5.7	92.8	1.5
T071202-01@19	91	58	2	0.64	1.14	0.09900	5.12	0.0153	1.68	0.33	95.9	4.7	97.8	1.6
T071202-02@1	101	130	2	1.28	3.60	0.09332	5.01	0.0147	2.07	0.41	90.6	4.4	93.8	1.9
T071202-02@2	204	108	3	0.53	1.08	0.09253	4.23	0.0140	1.61	0.38	89.9	3.6	89.4	1.4
T071202-02@3	115	150	2	1.31	14.86	0.09268	26.63	0.0142	1.81	0.07	90.0	23.2	90.7	1.6
T071202-02@4	290	361	6	1.25	3.18	0.09255	3.22	0.0143	1.60	0.50	89.9	2.8	91.3	1.4
T071202-02@5	91	111	2	1.22	4.10	0.09195	5.40	0.0144	1.66	0.31	89.3	4.6	92.4	1.5
T071202-02@6	174	307	4	1.76	6.24	0.08494	4.97	0.0141	1.58	0.32	82.8	4.0	90.5	1.4
T071202-02@7	152	137	3	0.90	2.16	0.08840	4.18	0.0139	1.50	0.36	86.0	3.5	88.9	1.3
T071202-02@8	77	142	2	1.85	5.91	0.09091	6.10	0.0139	1.88	0.31	88.4	5.2	88.7	1.7
T071202-02@9	116	161	2	1.39	3.93	0.09354	4.82	0.0144	1.88	0.39	90.8	4.2	92.0	1.7
T071202-02@10	54	40	1	0.74	4.58	0.09916	6.73	0.0144	1.77	0.26	96.0	6.2	92.2	1.6
T071202-02@11	110	139	2	1.27	5.73	0.08891	4.90	0.0145	2.00	0.41	86.5	4.1	92.5	1.8
T071202-02@12	96	123	2	1.28	3.82	0.09264	5.75	0.0142	1.70	0.30	90.0	5.0	90.8	1.5
T071202-02@13	174	226	4	1.30	2.33	0.09586	3.63	0.0142	1.63	0.45	92.9	3.2	90.8	1.5
T071202-02@14	55	46	1	0.83	5.45	0.09940	6.12	0.0149	1.75	0.29	96.2	5.6	95.4	1.7
T071202-02@15	130	176	3	1.35	4.06	0.08964	4.14	0.0143	1.59	0.38	87.2	3.5	91.4	1.4
T071202-02@16	168	241	4	1.43	2.89	0.09582	5.45	0.0143	1.56	0.29	92.9	4.9	91.8	1.4
T071202-02@17	134	164	3	1.22	4.59	0.08850	4.27	0.0138	1.68	0.39	86.1	3.5	88.7	1.5
T071202-02@18	189	346	4	1.83	10.25	0.09950	6.77	0.0145	1.80	0.27	96.3	6.2	92.5	1.7
T071202-02@19	102	129	2	1.27	4.43	0.09700	4.52	0.0142	1.65	0.36	94.0	4.1	91.2	1.5
T071203-01@1	238	297	5	1.25	7.04	0.09472	8.71	0.0139	1.64	0.19	91.9	7.7	88.7	1.4
T071203-01@2	98	124	2	1.26	2.34	0.09623	6.63	0.0144	1.64	0.25	93.3	5.9	92.1	1.5
T071203-01@3	199	241	4	1.21	3.44	0.08213	4.11	0.0142	1.55	0.38	80.1	3.2	90.7	1.4
T071203-01@4	103	258	3	2.52	8.59	0.08735	6.34	0.0140	1.64	0.26	85.0	5.2	89.7	1.5
T071203-01@5	100	140	2	1.40	2.73	0.09219	5.05	0.0145	1.83	0.36	89.5	4.3	92.8	1.7
T071203-01@6	143	203	3	1.42	1.79	0.09094	4.23	0.0147	1.72	0.41	88.4	3.6	93.9	1.6
T071203-01@7	191	222	4	1.16	2.05	0.09434	3.71	0.0144	1.66	0.45	91.5	3.3	92.1	1.5
T071203-01@8	184	202	4	1.10	1.33	0.09603	3.86	0.0145	1.59	0.41	93.1	3.4	93.0	1.5
T071203-01@9	143	362	4	2.54	5.31	0.08914	5.03	0.0145	1.61	0.32	86.7	4.2	92.6	1.5
T071203-01@10	215	246	4	1.14	1.75	0.10630	3.26	0.0144	1.57	0.48	102.6	3.2	92.1	1.4
T071203-01@11	174	235	4	1.35	3.73	0.08147	6.65	0.0141	1.58	0.24	79.5	5.1	90.1	1.4
T071203-01@12	147	152	3	1.04	2.00	0.09294	4.39	0.0146	1.53	0.35	90.2	3.8	93.3	1.4
T071203-01@13	116	161	3	1.39	3.55	0.10134	4.87	0.0152	1.66	0.34	98.0	4.6	97.5	1.6
T071203-01@14	110	140	2	1.27	5.25	0.09491	7.27	0.0142	1.69	0.23	92.1	6.4	90.7	1.5
T071203-01@15	98	86	2	0.88	2.61	0.09644	6.14	0.0145	1.57	0.26	93.5	5.5	92.6	1.4
T071203-01@16	93	77	2	0.83	3.72	0.09741	7.02	0.0150	1.86	0.27	94.4	6.3	95.8	1.8
T071203-01@17	106	145	2	1.37	6.16	0.08797	5.82	0.0145	1.65	0.28	85.6	4.8	92.9	1.5
T071203-01@18	110	113	2	1.03	0.00	0.09842	4.15	0.0143	1.58	0.38	95.3	3.8	91.6	1.4
T071203-01@19	103	133	2	1.30	2.97	0.08946	5.28	0.0143	1.66	0.31	87.0	4.4	91.7	1.5

Note: f_{206} is the proportion of common ^{206}Pb in total measured ^{206}Pb . ρ is the correlation coefficient.

a minority has inherited cores or metamict structures. All the analyses were conducted on portions of zircon with oscillatory zoning, which is interpreted as reflecting growth in magma. We report here 136 U–Pb zircon analyses from the six tuff beds in the Tiantai Basin (Table 1 and Fig. 4).

4.1. Sample T071201-01

Twenty five analyses of 25 zircons from sample T07120101-01 were obtained in sets of seven scans during a single analytical session. U and Th concentrations are relatively low. Th concentrations

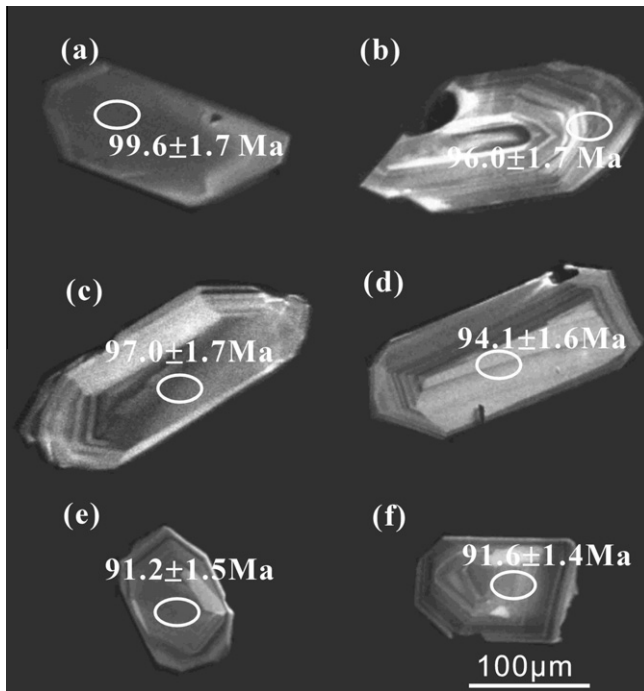


Fig. 3. Cathodoluminescence (CL) images of representative zircons. The ellipses on the analyzed zircon grains show the positions of U–Pb analytical sites. (a) T071201-01; (b) T071201-04; (c) T071201-05; (d) T071202-01; (e) T071202-02; (f) T071203-01. See Fig. 2 for sections and stratigraphic positions of the samples.

range from 15 to 174 ppm, U from 10 to 125 ppm, and Th/U ratios vary between 1.16 and 2.01. Common Pb contents in this sample is relatively high (Table 1). However it makes no significant difference to the result. Twenty-four of 25 analyses have indistinguishable $^{206}\text{Pb}/^{238}\text{U}$ isotopic ratios within analytical uncertainties. The spot three has distinguishable U–Pb isotopic compositions within analytical errors, giving a younger $^{206}\text{Pb}/^{238}\text{U}$ age of 91.4 ± 1.9 Ma. A Concordia age is calculated at 99.4 ± 1.1 Ma (MSWD = 0.2) based on 24 analyses (Fig. 4a), and weighted mean $^{206}\text{Pb}/^{238}\text{U}$ age is 99.4 ± 1.5 Ma, which is interpreted as the crystallization age of sample T071201-01.

4.2. Sample T071201-04

Twenty five analyses of 25 zircons from sample T071201-04 were obtained in sets of seven scans during a single analytical session. Th concentrations range from 57 to 1767 ppm, U from 51 to 1572 ppm, and Th/U ratios vary between 0.66 and 2.06. Common Pb is low, with f_{206} values mostly <1%. The concordant 25 analyses have indistinguishable U–Pb isotopic compositions within analytical errors, a Concordia age is calculated at 97.2 ± 0.7 Ma (MSWD = 0.7) (Fig. 4b), and weighted mean $^{206}\text{Pb}/^{238}\text{U}$ age is 97.2 ± 0.8 Ma, which is interpreted as the crystallization age of sample T071201-04.

4.3. Sample T071201-05

Twenty nine analyses of 29 zircons from sample T071201-05 were obtained in sets of seven scans during a single analytical session. They have relatively constant abundances of Th and U, with Th concentrations ranging from 13 to 302 ppm, U from 18 to 362 ppm, and Th/U ratios varying between 0.53 and 1.69. Common Pb is low, with f_{206} values mostly <1%. For the 29 analyses, $^{206}\text{Pb}/^{238}\text{U}$ ratios agree internally within the analytical precision, a Concordia age is calculated at 96.1 ± 0.9 Ma (MSWD = 2.2)

(Fig. 4c), and weighted mean age is 96.2 ± 1.0 Ma, which is interpreted as the crystallization age of sample T071201-05.

4.4. Sample T071202-01

Nineteen analyses of 19 zircons from sample T071202-01 were obtained in sets of seven scans during a single analytical session. They have relatively constant abundances of Th and U, with Th concentrations ranging from 58 to 295 ppm, U from 70 to 379 ppm, and Th/U ratios varying between 0.64 and 1.79. Common Pb is low. All of the 19 analyses have indistinguishable U–Pb isotopic compositions within analytical errors, a Concordia age is calculated at 94.4 ± 0.7 Ma (MSWD = 0.4) (Fig. 4d), and weighted mean $^{206}\text{Pb}/^{238}\text{U}$ age is 94.4 ± 0.9 Ma, which is interpreted as the crystallization age of sample T071202-01.

4.5. Sample T071202-02

Nineteen analyses of 19 zircons from sample T071202-02 were obtained in sets of seven scans during a single analytical session. They have relatively constant abundances of Th and U, with Th concentrations ranging from 40 to 346 ppm, U from 54 to 290 ppm, and Th/U ratios varying between 0.53 and 1.85. Common Pb of the spot 3 is high, and the others are low. Rejecting the spot 3, the remaining 18 analyses have indistinguishable U–Pb isotopic compositions within analytical errors, a Concordia age is calculated at 91.1 ± 0.7 Ma (MSWD = 2.8) (Fig. 4e), and weighted mean $^{206}\text{Pb}/^{238}\text{U}$ age is 91.2 ± 0.9 Ma, which is interpreted as the crystallization age of sample T071202-02.

4.6. Sample T071203-01

Nineteen analyses of 19 zircons from sample T071203-01 were obtained in sets of seven scans during a single analytical session. They have relatively constant abundances of Th and U, with Th concentrations ranging from 77 to 362 ppm, U from 93 to 238 ppm, and Th/U ratios varying between 0.83 and 2.54.

Eighteen of the 19 analyses have indistinguishable $^{206}\text{Pb}/^{238}\text{U}$ isotopic ratios within analytical uncertainties. The spot 13 yield significantly old $^{206}\text{Pb}/^{238}\text{U}$ age of 97.4 ± 1.6 Ma, most likely to be xenocryst. Rejecting this spot, the remaining 18 concordant analyses give a Concordia age of 91.9 ± 1.0 Ma (MSWD = 3.9) (Fig. 4f), and weighted mean $^{206}\text{Pb}/^{238}\text{U}$ age is 91.9 ± 0.7 Ma, which is interpreted as the crystallization age of sample T071203-01.

5. Discussion and conclusions

Despite abundant vertebrate fossil discoveries from the middle Cretaceous of Zhejiang Province, the age of the fossil-bearing deposits remains largely unresolved. Our dating results suggest that the ages of the Lajia and Chichengshan formations are about 96–99 Ma (Cenomanian) and 91–94 Ma (Turonian), respectively, confirming previous estimates that they belong to the early Late Cretaceous.

Dinosaur eggs or fragments are abundant and extensively distributed in China. They can be very informative in biostratigraphic division and correlation of continental strata where other fossils are relatively lacking. Recently, there are also important discoveries of invertebrate fossil evidences in both Xixia and Nanxiong Basins, which make it possible to correlate the dinosaur egg bearing sequences with other Upper Cretaceous non-marine sequences in northern and southern China (Li et al., 2009a,b, 2010a,b). Based on the spinicaudatan Tylestheria, Li et al. (2009a,b) suggest that dinosaur eggs in the Upper Cretaceous Majiacun Formation, Xixia Basin, China are of Turonian age. Wang et al. (2012) provided a

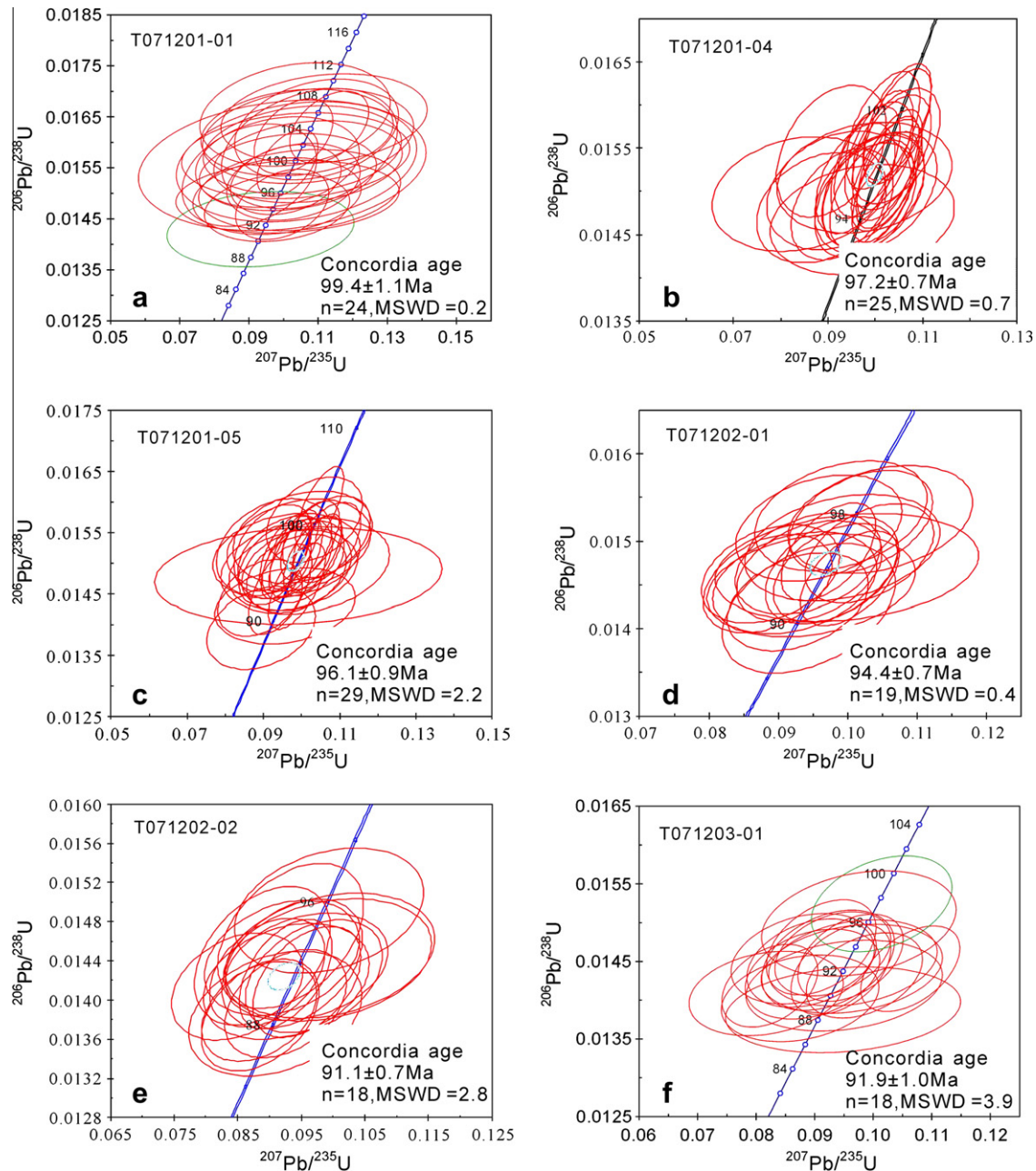


Fig. 4. U–Pb Concordia diagrams showing SIMS analytical data for zircons from the tuff layers. Data-point error ellipses are 2σ . Green data-points were not included in the age calculation.

preliminary summary of the correlation among major Chinese dinosaur egg faunas and their stratigraphy, and argued, mainly based on lithostratigraphy and dinosaur egg biostratigraphy, that the Tiantai Basin contains the earliest Late Cretaceous oofauna in China, followed successively by those from the Xixia, Laiyang and Nanxiong basins. The dinosaur egg assemblage (such as the presence of dictyoolithids and faveoolithids, but lacking of spheroolithids and ovaloolithids) from the Tiantai Basin was also regarded as more primitive than those from other Late Cretaceous basins (Zhao, 1993; Wang et al., 2012), which is consistent with our result of the early Late Cretaceous age for the Laijia and Chichengshan formations.

Although our results provide precise geochronological evidence and a time frame for the earliest Late Cretaceous dinosaur egg fauna in China, we believe that establishing a complete and precise time frame for the Late Cretaceous terrestrial deposits in China will

definitely have important implications not only for further correlations of Chinese dinosaur egg faunas with those from other regions, but also for the discussion of the origin and evolution of various dinosaur egg taxa.

Acknowledgments

We thank Jian Zhang and Gui-Feng Wang (Tiantai Museum, Zhejiang Province, China), Li-Jun Zhao (Zhejiang Museum of Natural History, Zhejiang Province, China), Wei Gao, Yan Li and Shukang Zhang (Institute of Vertebrate Paleontology and Paleoanthropology, Chinese Academy of Sciences) for help in the field. We gratefully acknowledge the careful and constructive comments of Ian Williams and an anonymous reviewer, which considerably improved the manuscript. This research was supported by the National Natural Science Foundation of China (40772017, 40825005,

41172018, 90714010, 40821091 and 40925012), National Key Basic Research Program of China (973 Program 2012CB821900).

References

- Black, L.P., Kamo, S.L., Allen, C.M., Aleinikoff, J.N., Davis, D.W., Korsch, R.J., Foudoulis, C., 2003. TEMORA 1: a new zircon standard for Phanerozoic U–Pb geochronology. *Chemical Geology* 200, 155–170.
- Chang, M., Chow, J., 1977. On Late Mesozoic fossil fishes from Zhejiang Province, China. *Memoirs of the Institute of Vertebrate Paleontology and Paleoanthropology Academia Sinica* 12, 1–59.
- Fang, X., Wang, Y., Jiang, Y., 2000. On the Late Cretaceous fossil eggs of Tiantai, Zhejiang. *Geological Review* 46, 105–112.
- Fang, X., Lu, L., Jiang, Y., Yang, L.F., 2003. Cretaceous fossil eggs from the Tiantai Basin of Zhejiang, with a discussion on the extinction of dinosaurs. *Geological Bulletin of China* 22, 512–520 (in Chinese).
- Ireland, T.R., Williams, I.S., 2003. Considerations in Zircon Geochronology by SIMS. *Reviews in Mineralogy and Geochemistry* 53, 215–241.
- Jin, X., Azuma, Y., Jackson, F.D., Varricchio, D.J., 2007. Giant dinosaur eggs from the Tiantai basin, Zhejiang Province, China. *Canadian Journal of Earth Sciences* 44, 81–88.
- Li, G., Chen, P., Wang, D., Batten, D.J., 2009a. The spinicaudatan *Tylestheria* and biostratigraphic significance for the age of dinosaur eggs in the Upper Cretaceous Majiacun Formation, Xixia Basin, Henan Province, China. *Cretaceous Research* 30, 477–482.
- Li, X.H., Liu, Y., Li, Q.L., Guo, C.H., Chamberlain, K.R., 2009b. Precise determination of Phanerozoic zircon Pb/Pb age by multicollector SIMS without external standardization. *Geochemistry, Geophysics, Geosystems*. <http://dx.doi.org/10.1029/2009GC002400>.
- Li, G., Hirano, H., Batten, D.J., Wan, X.Q., Willems, H., Zhang, X.Q., 2010a. Biostratigraphic significance of spinicaudatans from the Upper Cretaceous Nanxiong group in Guangdong, South China. *Cretaceous Research* 31, 387–395.
- Li, Q.L., Li, X.H., Liu, Y., Tang, G.Q., Yang, J.H., Zhu, W.G., 2010b. Precise U–Pb and Pb–Pb dating of Phanerozoic baddeleyite by SIMS with oxygen flooding technique. *Journal of Analytical Atomic Spectrometry* 25, 1107–1113.
- Luo, Y., Yu, Y., 2004. Age of the Yongkang Group and regional stratigraphic correlation. *Geology in China* 31, 395–399.
- Qian, M., Jiang, Y., Chen, R., Jiang, Y.G., Zhang, Y., Xin, G., 2008a. Late Cretaceous troodontid eggs from Tiantai, Zhejiang. *Acta Palaeontologica Sinica* 47, 248–255.
- Qian, M., Jiang, Y., Jiang, Y.G., Zhang, Y., Chen, R., Li, L., Xing, G., 2008b. New evidence on fossil eggs of Cretaceous Tyrannosaurs in eastern China. *Jiangsu Geology* 32, 86–97.
- Stacey, J.S., Kramers, J.D., 1975. Approximation of terrestrial lead isotope evolution by a two-stage model. *Earth and Planetary Science Letters* 26, 207–221.
- Steiger, R.H., Jaeger, E., 1977. Subcommittee on geochronology: convention on the use of decay constants in geo- and cosmochronology. *Earth and Planetary Science Letters* 36, 359–362.
- Wang, Q., 2010. A study of dinosaur eggs from the Upper Cretaceous Tiantai Basin, Zhejiang Province and the associated strata of other basins. In: PhD thesis, Institute of Vertebrate Paleontology and Paleoanthropology, and the Graduate University of Chinese Academy of Sciences, Beijing, pp. 1–89 (in Chinese).
- Wang, F., Yang, L.K., Wang, L., Shen, J.L., Xing, G.F., Chen, R., Pan, Y.X., Zhu, R.X., 2010a. The boundary ages of the late Mesozoic volcanic-sedimentary strata on South China: constrains from $^{40}\text{Ar}/^{39}\text{Ar}$ geochronology and paleomagnetism. *Science in China Series D: Earth Sciences* 40, 1552–1570 (in Chinese).
- Wang, Q., Wang, X., Zhao, Z., Jiang, Y., 2010b. New oogenus of Elongatoolithidae from the Upper Cretaceous Chichengshan Formation of Tiantai Basin, Zhejiang Province. *Vertebrata Palasiatica* 48, 111–118.
- Wang, Q., Zhao, Z., Wang, X., Jiang, Y., Zhang, S., 2010c. A new oogenus of Macroelongatoolithid eggs from the Upper Cretaceous Chichengshan formation of the Tiantai Basin, Zhejiang Province and a revision of the Macroelongatoolithids. *Acta Palaeontologica Sinica* 49, 73–86.
- Wang, Q., Zhao, Z., Wang, X., Jiang, Y., 2011. New ootypes of dinosaur eggs from the Late Cretaceous in Tiantai Basin, Zhejiang Province. *Vertebrata Palasiatica* 49, 446–449.
- Wang, X., Wang, Q., Jiang, S., Cheng, X., Zhang, J., Zhao, Z., Jiang, Y., 2012. Dinosaur egg faunas of the Upper Cretaceous terrestrial red beds of China and their stratigraphical significance. *Journal of Stratigraphy* 36, 400–416.
- Wiedenbeck, M., Allé, P., Corfu, F., Griffin, W.L., Meier, M., Oberli, F., Von Quadt, A., Roddick, J.C., Spiegel, W., 1995. Three natural zircon standards for U–Th–Pb, Lu–Hf, trace element and REE analyses. *Geostandards Newsletter* 19, 1–23.
- Xu, X., Wang, K., Zhao, X., Sullivan, C., Chen, S., 2010. A new leptoceratopsid (Ornithischia: Ceratopsia) from the Upper Cretaceous of Shandong, China and its implications for neoceratopsian evolution. *PLoS ONE*. <http://dx.doi.org/10.1371/journal.pone.0013835>.
- Yu, Y., Jin, X., Wu, X., Zhang, Z., 2010. The epochs of dinosaurs and fossil eggs from Zhejiang Province. *Journal of Stratigraphy* 37, 94–100.
- Yu, F., Wang, L., Du, L., Zou, X., Wang, G., Zhang, H., 2012. Distribution of dinosaurs and their egg fossils in the Cretaceous basins, Zhejiang Province. *Journal of Stratigraphy* 36, 77–88.
- Zhang, S., 2010. A parataxonomic revision of the Cretaceous faveoolithid eggs of China. *Vertebrata Palasiatica* 48, 203–219.
- Zhao, Z., 1993. Structure formation and evolutionary trends of dinosaur eggshells. In: Kobayashi, I., Mutvei, H., Sahni, A. (Eds.), *Structure, Formation and Evolution of Fossil Hard Tissues*. Tokai University Press, Tokyo, pp. 195–212.
- Zhao, Z., 1994. Dinosaur eggs in China: on the structure and evolution of eggshells. In: Carpenter, K., Hirsch, K.F., Horner, J.R. (Eds.), *Dinosaur Eggs and Babies*. Cambridge University Press, Cambridge, pp. 184–203.
- Zhao, Z., Mao, X., Chai, Z., Yang, G., Zhang, F., Yan, Z., 2009. Geochemical environmental changes and dinosaur extinction during the Cretaceous–Paleogene (K/T) transition in the Nanxiong Basin, South China: evidence from dinosaur eggshell. *Chinese Science Bulletin* 54, 806–815.