

## 1 Supporting Information

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### Detailed descriptions for Analytical Methods

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5 Chemical and isotopic determinations were carried out via several different analysis methods in  
6 different independent laboratories to identify the utility of SA02 as a zircon reference material  
7 (Table 1). Trace element analysis was achieved using LA-ICP-MS. U-Pb dataset were produced  
8 by (CA)-ID-TIMS, SIMS and LA-ICP-MS. Laser fluorination and SIMS were used for measuring  
9 O isotopic compositions. As for Hf isotope, solution and laser ablation MC-ICP-MS were adopted  
10 for the ratio determinations. Full details of the analytical procedures have previously been reported  
11 <sup>1-12</sup>, and analytical protocols are given here in brief.

12 **Table 1.** Summary of the methods employed on SA02 zircon crystal.

Sample	Trace element	U-Pb dating			Hf isotope		O isotope	
		(CA)-ID-TIMS	SIMS	LA-ICP-MS	Solution MC-ICP-MS	LA MC-ICP-MS	SIMS	Laser fluorination
SA02	IGGCAS	BGS TJCGS	IGGCAS	NWU CUG(WuHan) IGGCAS	IGGCAS	NWU IGGCAS	IGGCAS	USTC IGGCAS

13 BGS: British Geological Survey, U.K.

14 TJCGS: The Tianjin Centre, China Geological Survey (China)

15 IGGCAS: The Institute of Geology and Geophysics, Chinese Academy of Sciences (China)

16 NWU: The Northwest University (China)

17 USTC: The University of Science and Technology of China (China)

18 CUG (WuHan): The China University of Geosciences, Wuhan (China)

### 19 1 Trace Element determinations

20 Trace element analysis of the SA02 zircon were carried out using an Agilent 7500a quadrupole  
21 ICP-MS coupled with a Coherent Geolas HD 193 nm excimer laser ablation system at IGGCAS  
22 (Table 2). Helium gas was utilized as the carrier gas and mixed with argon gas downstream from  
23 the ablation cell. A repetition rate of 5 Hz, a spot size of 44  $\mu\text{m}$  and a fluence of 5 J/cm<sup>2</sup> were  
24 adopted for the laser in the process of the measurement. Each spot analysis consists of approximate  
25 20 s of background and 60 s of ablation. ARM-1 glass was used as the standard for the calibration  
26 of relative element sensitivities to achieve quantitative results of trace element <sup>12</sup>. The Glitter  
27 software was used for the data reduction <sup>13</sup>. More details for the analytical procedures are provided  
28 by Xie et al.<sup>4</sup>.

29 **Table 2.** Typical solution/LA-ICP-MS instrument parameters for U-Pb dating, trace element and  
30 Hf isotopic compositions of SA02 zircon

MC-ICP-MS cup configuration									
Cup	L4	L3	L2	L1	C	H1	H2	H3	H4
Hf	<sup>172</sup> Yb	<sup>173</sup> Yb	<sup>175</sup> Lu	<sup>176</sup> Hf	<sup>177</sup> Hf	<sup>178</sup> Hf	<sup>179</sup> Hf	<sup>180</sup> Hf	<sup>182</sup> W
Instrumentations									
Mass spectrometry		Thermo Fisher Neptune Plus (MC-ICP-MS)				Agilent 7500a (ICP-Q-MS)			
RF forward power (W)		~1200				~1400			
Interface cones		Nickel Standard Sampler cones and “H” Skimmer cones				Nickel cones			
Integration times		0.131s for laser Hf isotope; 4.191s for solution Hf isotope				6ms for Ti, Hf and REE; 10ms for <sup>232</sup> Th, <sup>238</sup> U; 15ms for <sup>204</sup> Pb, <sup>206</sup> Pb, <sup>208</sup> Pb; 30ms for <sup>207</sup> Pb			
Background/baseline		0s				20s			
Carrier gas (L/min)		~ 0.95				~ 0.85			

<b>Laser ablation system</b>	<b>Geolas Pro/HD</b>
Laser system	Compex 102, ArF excimer UV 193nm
Fluence	~ 5 J/cm <sup>2</sup> for trace element, U-Pb dating and Hf isotope
Spot size	44 μm for trace element and U-Pb dating; 32 μm for Hf isotope
Ablation duration	60s for trace element and U-Th-Pb dating; 26s for Hf isotope
Sampling mode/Repetition rate	Static spot ablation; 5 Hz for trace element and U-Pb dating, 6 Hz for Hf isotope
Sample preparation	Conventional mineral separation, 1inch resin mount
Imaging	Transmissive light, reflected light and CL imaging,
<b>Data processing</b>	
Reference material information	Trace element: ARM-1 glass is used as reference material U-Pb dating :91500 is used as primary reference material, and SA 01 is used as secondary standard for validation Hf isotope: Mud Tank is used as the primary standard for validation; SA 01 is used for the monitor standard.
Data processing package used	For trace elements and U-Pb data processing, Glitter software was used for isotopic and elemental fractionation, instrumental mass bias calibration. For Hf isotope, an in-house Microsoft Excel macro written in VBA (Visual Basic for Applications) was used for mass fraction correction, interference correction and uncertainty propagation
Common Pb correction	No common-Pb correction applied to data.
Uncertainty Level	Ages are quoted at 2s absolute unless otherwise stated.

## 31 **2 U-Pb geochronology**

### 32 **2.1 CA-ID-TIMS (BGS)**

33 Two SA02 zircon shards were first annealed at 900 °C for 60 h in a muffle furnace. Subsequently,  
34 individual shards were loaded into Teflon™ microcapsules with 100 μl of a ca. 5:1 mixture of 29  
35 mol l<sup>-1</sup> HF and 30% HNO<sub>3</sub> in which crystals were chemically abraded at 210 °C for 12 h. After  
36 chemical abrasion, residual crystal fragments were rinsed in 6 mol l<sup>-1</sup> HCl and 30% HNO<sub>3</sub>  
37 solutions.

38 Cleaned crystals were spiked with ET2535 tracer solution and dissolved at 220 °C for 60 h using  
39 120 μl of 29 mol l<sup>-1</sup> HF with a trace amount of 30% HNO<sub>3</sub>. The solutions were then dried down  
40 and dissolved in 3M HCl at 180°C for ~12 hours to convert the samples to chloride form, after  
41 which U and Pb were purified from the dissolved sample by anion-exchange column chemistry. U–  
42 Pb isotope measurements were performed on a Thermo Electron Triton instrument equipped with  
43 an ion-counting SEM system, in single ion-counter (peak-hopping) mode for monoatomic Pb and  
44 multiple Faraday detector mode for U-oxide at BGS. Data reduction, age calculation and  
45 uncertainty propagation were performed using Tripoli and ET\_Redux software <sup>14, 15</sup>. A magma  
46 Th/U ratio of 2.8 was used for <sup>230</sup>Th disequilibrium correction and a <sup>238</sup>U/<sup>235</sup>U ratio of 137.818 ±  
47 0.045 was adopted in data reduction <sup>16</sup>.

### 48 **2.2 ID-TIMS (TJCGS)**

49 The ID-TIMS analysis for SA02 zircon were performed at the TJCGS. Several SA02 zircon shards  
50 were microscopically selected based on clarity and apparent absence of inclusions or fractures.  
51 Before the analysis, acids used in the experiments were completely purified three times, and a  
52 Milli-Q system offers ultrapure water during the measurement. The zircon shards were  
53 successively washed in 1 mol l<sup>-1</sup> HCl, 1 mol l<sup>-1</sup> HNO<sub>3</sub> and 1 mol l<sup>-1</sup> HF solutions at 100 °C for 2h,  
54 respectively. Cleaned crystals were dissolved in concentrated HF at 185 °C for 72h. the digested  
55 sample solutions were divided into two aliquots, one aliquot for Pb isotopic ratio measurements  
56 (ca. 65%) and the other for U-Pb contents determinations (ca. 35%) added five drops (ca.38 mg)  
57 of a mixed <sup>208</sup>Pb-<sup>235</sup>U spike. Two solution aliquots were weighed accurately and then evaporated  
58 to dryness. Lead and U were separated using AG1 × 8 anion exchange resin with 6 mol l<sup>-1</sup> HCl for

59 Pb isotope and 7 mol l<sup>-1</sup> HNO<sub>3</sub> for U isotope. U-Pb isotopic ratios were measured on a Thermo  
60 Fisher Triton TIMS instrument with H<sub>3</sub>PO<sub>4</sub> and silica gel onto degassed Re single filaments. Total  
61 procedural blanks in the process of measurements were 10-27 pg for Pb and 1-3 pg for U,  
62 respectively. IsoplotR<sup>17, 18</sup> software was employed for the date reduction. Common Pb was  
63 monitored using <sup>204</sup>Pb, and corrections were made based on the blank Pb isotopic composition and  
64 initial Pb isotopic composition given by the model of Stacey and Kramers<sup>19</sup>.

### 65 2.3 SIMS (IGGCAS)

66 Measurements of U-Pb ages were conducted using CAMECA IMS 1280HR SIMS at IGGCAS  
67 following the published method<sup>5</sup>. A O<sub>2</sub><sup>-</sup> primary beam of ca. 10 nA accelerated at -13 kV and an  
68 ellipsoidal spot of ca. 20 μm × 30 μm was adopted for the SIMS. An energy window of 60 eV and  
69 a mass resolution of ca. 5400 (at 10% peak height) were used to measure Pb ion peaks with isobaric  
70 interferences removal. Secondary ion beam signals were collected with a single electron multiplier  
71 on ion-counting mode by peak jumping sequences. Each measurement involves seven cycles.  
72 Obtained Pb/U ratios were calibrated relative to the zircon reference material Plešovice (<sup>206</sup>Pb/<sup>238</sup>U  
73 age = 337 Ma) using a sample <sup>238</sup>U/<sup>235</sup>U ratio of 137.818 ± 0.045<sup>16, 20</sup>. For the uncertainty of single  
74 analytical spots, a long-term uncertainty of 1.5% (1s RSD) for <sup>206</sup>Pb/<sup>238</sup>U measurements of the  
75 Plešovice zircon reference material was propagated<sup>5</sup>. The common lead correction was achieved  
76 using non-radiogenic <sup>204</sup>Pb and the data were processed using IsoplotR<sup>17, 18</sup>. Uncertainties of data  
77 on individual analyses are reported at the 1s level. Moreover, the ‘external reproducibility’ of the  
78 SIMS U-Pb analysis was assessed on the in-house zircon reference material (Qinghu) analyzed as  
79 an unknown together with other unknown zircons, giving a mean <sup>206</sup>Pb/<sup>238</sup>U age of 159.0 ± 3.0 Ma  
80 (2s, n = 7) consistent with the reported value (159.45 ± 0.16 Ma)<sup>5</sup>.

### 81 2.4 LA-ICP-MS (NWU)

82 The LA-ICP-MS U-Pb dating for SA02 zircon at NWU was performed using an Agilent 7900 ICP-  
83 MS coupled with a RESOLution M-50 193 nm laser system equipped with a two-volume laser  
84 ablation cell, a Squid smoothing device and a computer-controlled high-precision X-Y stage. The  
85 characteristic of two-volume cell comprises the capability of avoiding cross contamination and  
86 reducing background flushing time, while the Squid smoothing device could give a smooth signal  
87 with laser pulse rates down to 1 Hz. Helium was used as carrier during laser ablation and it entered  
88 the cell body at its bottom to fill the big cell. Helium from both bottom and top through the funnel  
89 cell entrained the sample aerosol and argon was admixed downstream, in front of the squid signal  
90 smoothing device, into the ICP-MS. Stability and sensitivity of the ICP-MS was optimized in  
91 solution mode before connected to a laser ablation system. The plasma was kept lighted during  
92 removing the spray chamber and connecting the laser ablation system. Line-scan mode was  
93 utilized to tune the operating parameters of ICP-MS using NIST SRM 610 glass. The nebulizer  
94 gas flow rate and the ion optics volts were optimized to maximize the sensitivity and to make sure  
95 the <sup>232</sup>Th<sup>16</sup>O<sup>+</sup>/<sup>232</sup>Th<sup>+</sup> ratios below 0.2% and the ratios of <sup>238</sup>U/<sup>232</sup>Th among 0.96-1.00. A repetition  
96 rate of 5 Hz, a spot size of 43 μm and a fluence of 6 J/cm<sup>2</sup> were adopted for the laser. All  
97 measurements for isotopes of <sup>238</sup>U, <sup>232</sup>Th, <sup>208</sup>Pb, <sup>207</sup>Pb, <sup>206</sup>Pb, <sup>204</sup>Pb, and <sup>202</sup>Hg were collected in  
98 peak-jumping mode. Mass 204 was measured to monitor the presence of common Pb in zircon  
99 grains and <sup>202</sup>Hg was measured in order to correct for the isobaric interference of <sup>204</sup>Hg on <sup>204</sup>Pb.  
100 Zircon standard material 91500 was used as an external standard to calibrate the zircon U-Pb ages.  
101 Data reduction was performed using ICPMSDataCal software, and the results were normalized

102 according to 91500 <sup>7, 21-23</sup>. Zircon reference material Plešovice was analyzed as an unknown  
103 sample to monitor the accuracy and reproducibility of the measurement, which yielded a mean  
104 <sup>206</sup>Pb/<sup>238</sup>U age of 338 ± 5 Ma (2s, n = 7) consistent with the reported value <sup>20</sup>.

## 105 **2.5 LA-ICP-MS (CUG (Wuhan))**

106 U-Pb age determinations of the SA02 zircon were carried out using Agilent 7500a ICP-MS  
107 coupled with GeoLas 2005 193 nm laser ablation system at the State Key Laboratory of Geological  
108 Processes and Mineral Resources, China University of Geosciences, Wuhan following proposed  
109 methods <sup>7, 8, 21</sup>, and a brief description is offered here. A “wire” signal smoothing device is used  
110 during the measurements, by which smooth signals are achieved even at very low laser repetition  
111 rates down to 1 Hz <sup>24</sup>. Argon was used as the make-up gas and mixed with the carrier gas of helium  
112 via a T-connector before entering the ICP adding Nitrogen to decrease the detection limit and  
113 improve precision <sup>8, 25</sup>). The data process was performed by ICPMSDataCal <sup>7, 21</sup>. Zircon reference  
114 material 91500 was used as external standard for U-Pb dating. Uncertainty of preferred values for  
115 zircon reference material 91500 was propagated to the ultimate results of the samples <sup>22</sup>. Concordia  
116 diagrams and weighted mean calculations were made using IsoplotR <sup>17, 18</sup>. Zircon reference  
117 material GJ-1 was analyzed together with SA02 zircon as an unknown to assess the ‘external  
118 uncertainties’ of measurements by LA-ICP-MS, which gave a mean <sup>206</sup>Pb/<sup>238</sup>U age of 601 ± 8 Ma  
119 (2s, n = 13) consistent with the published value <sup>26</sup>.

## 120 **2.6 LA-ICP-MS (IGGCAS)**

121 Analysis of U-Pb age was conducted by LA-ICP-MS at IGGCAS using the same system as for  
122 trace element determinations of SA02 zircon and identical instrument parameters reported by Xie  
123 et al.<sup>4</sup> (Table 2). Zircon reference material 91500 was used as the primary reference material for  
124 the correction of depth-dependent elemental and isotopic fractionation, mass discrimination and  
125 drift <sup>22</sup>. Software program ICPMSDataCal 9.0 was used for the data process <sup>7</sup>. The accuracy and  
126 reproducibility of the analyses were assessed by measurements of zircon reference material SA01,  
127 which gave a mean <sup>206</sup>Pb/<sup>238</sup>U age of 535 ± 7 Ma (2s, n = 9) consistent with the reported value <sup>27</sup>.  
128 The age calculations and plotting of Concordia diagrams were performed using IsoplotR <sup>17, 18</sup>.

## 129 **3 Oxygen isotope determination**

### 130 **3.1 Laser fluorination (USTC and IGGCAS)**

131 Oxygen isotope analysis of SA02 zircon using laser fluorination method were carried out in two  
132 laboratories, USTC and IGGCAS with the use of a Finnegan Delta XP mass spectrometer and a  
133 Thermo Scientific Fisher M252 mass spectrometer, respectively <sup>28, 29</sup>. O<sub>2</sub> was extracted utilizing a  
134 25 W CO<sub>2</sub> laser (λ = 10.6 mm) and BrF<sub>5</sub> reagent for the <sup>18</sup>O/<sup>16</sup>O ratios measurement normalized  
135 to VSMOW (Vienna Standard Mean Ocean Water) and expressed on the δ<sup>18</sup>O-scale <sup>1, 30</sup>. The  
136 ‘external reference materials’ of 91500 zircon (δ<sup>18</sup>O = 9.86 ‰) and the 04BXL07 garnet (δ<sup>18</sup>O =  
137 3.70 ‰) were used for the data correction, respectively <sup>1, 31</sup>.

## 138 3.2 SIMS (IGGCAS)

139 Determinations of oxygen isotopes were conducted using CAMECA IMS 1280 SIMS at IGGCAS.  
140 The analytical procedure was reported by Li et al.<sup>6</sup>. A Cs<sup>+</sup> primary beam of ca. 1.5 nA and a  
141 diameter of ca. 10mm were adopted for the SIMS. A normal electron gun was used to compensate  
142 the charge effect generated by bombarding the area. Two isotopes, <sup>16</sup>O and <sup>18</sup>O, were measured  
143 simultaneously. Zircon reference material Penglai ( $\delta^{18}\text{O} = 5.31\text{‰}$ ) was used as the primary  
144 reference material for data calibration using conventional standard-sample bracketing. Other  
145 zircon reference material Qinghu was measured as a quality control reference material, giving a  
146 mean of  $5.32 \pm 0.39\text{‰}$  (2s, n = 14) consistent with the reported value <sup>10</sup>.

## 147 4 Hf isotope determination

### 148 4.1 Solution MC-ICP-MS (IGGCAS)

149 The solution analysis of Hf isotope for SA02 zircon presented here were carried out at the  
150 IGGCAS. Full details of the dissolution and sequential extraction of Hf has previously been  
151 reported by Yang et al.<sup>9</sup>. A brief introduction is offered here. Fifteen zircon shards were dissolved  
152 in concentrated HF-HNO<sub>3</sub> at 220 °C for 3 days, followed by evaporated to dryness and re-dissolved  
153 in 3 mol l<sup>-1</sup> HCl. A cation-exchange resin column was used to separate and purify Hf before  
154 analysis. Obtained samples were analyzed on a Thermo Scientific Fisher Neptune plus MC-ICP-  
155 MS. Instrumental mass bias was corrected using a <sup>179</sup>Hf/<sup>177</sup>Hf ratio of 0.7325 by the exponential  
156 law. Measured <sup>173</sup>Yb and <sup>175</sup>Lu values were used to correct the possible isobaric interferences of  
157 <sup>176</sup>Yb and <sup>176</sup>Lu on <sup>176</sup>Hf, applying <sup>176</sup>Lu/<sup>175</sup>Lu = 0.02655 and <sup>176</sup>Yb/<sup>173</sup>Yb = 0.79631 and assuming  
158 the Yb and Lu mass discrimination is the same as that of Hf <sup>32</sup>. The accuracy of analysis is  
159 demonstrated by repeat analyses of Alfa Hf solution (JMC14374), which provided a mean  
160 <sup>176</sup>Hf/<sup>177</sup>Hf value of  $0.282198 \pm 0.000013$  (2s, n = 23) identical to the reported value within  
161 uncertainties <sup>3,9</sup>.

### 162 4.2 LA MC-ICP-MS (NWU)

163 The Lu–Hf isotopic compositions of SA02 zircon were analyzed by using a Nu Plasma II MC-  
164 ICP-MS connected to a RESOLution M-50 193 nm laser system at NWU. Helium acted as a carrier  
165 gas mixed with argon downstream. A spot size of 44 μm, a repetition rate of 6 Hz and an energy  
166 density of 6 J/cm<sup>2</sup> were used for the laser ablation system. Ion signals of <sup>172</sup>Yb, <sup>173</sup>Yb, <sup>175</sup>Lu, <sup>176</sup>(Hf  
167 +Yb +Lu), <sup>177</sup>Hf, <sup>178</sup>Hf, <sup>179</sup>Hf and <sup>180</sup>Hf were detected simultaneously. The detail information of  
168 these instruments, analysis strategy and data deduction can be found in published literature <sup>33,34</sup>.  
169 Zircon reference material 91500 and Mud Tank were analyzed as an unknown sample to assess  
170 the quality of the data during the analysis. The obtained <sup>176</sup>Hf/<sup>177</sup>Hf ratios of the 91500 zircon and  
171 Mud Tank zircon were  $0.282293 \pm 0.000035$  (n=16, 2s) and  $0.282510 \pm 0.000033$  (n=8, 2s),  
172 respectively, which are consistent with the reported value within analytical uncertainty <sup>22,35</sup>.

### 173 4.3 LA MC-ICP-MS (IGGCAS)

174 The Lu–Hf isotopic analyses for SA02 zircon were carried out by LA-MC-ICP-MS at IGGCAS  
175 using Thermo Scientific Fisher Neptune plus MC-ICP-MS attached with a Coherent Geolas Pro  
176 193nm excimer laser ablation system. A laser spot size of 32μm or 44μm, a repetition rate of 6 Hz

177 and an energy density of *ca.* 5 J cm<sup>-2</sup> were set for the laser ablation system (Table 2). The carrier  
178 gas consisted of helium, argon and nitrogen. The detailed analytical procedures were similar to  
179 those described previously<sup>3</sup>. An in-house Microsoft Excel macro written in VBA (Visual Basic  
180 for Applications) was used for data process including mass fraction correction, interference  
181 correction and uncertainty propagation. Instrumental mass bias for the <sup>176</sup>Yb/<sup>177</sup>Hf, <sup>176</sup>Lu/<sup>177</sup>Hf and  
182 <sup>176</sup>Hf/<sup>177</sup>Hf ratios were normalized to the natural abundance ratio of <sup>179</sup>Hf/<sup>177</sup>Hf = 0.7325 using the  
183 exponential law. The mean <sup>173</sup>Yb/<sup>172</sup>Yb ratio of the individual spot was measured to calculate the  
184 fractionation coefficient ( $\beta_{Yb}$ ) and the contribution of <sup>176</sup>Yb to <sup>176</sup>Hf by applying the natural  
185 abundance ratios of <sup>176</sup>Yb/<sup>172</sup>Yb = 0.588673 and <sup>173</sup>Yb/<sup>172</sup>Yb = 0.73925. The isobaric interference  
186 of <sup>176</sup>Lu on <sup>176</sup>Hf was corrected by measuring the intensity of the interference-free <sup>175</sup>Lu isotope  
187 with a recommended <sup>176</sup>Lu/<sup>175</sup>Lu ratio of 0.02655 and assuming  $\beta_{Lu} = \beta_{Yb}$ <sup>3</sup>. Accuracy and external  
188 reproducibility of the analyses were controlled by repeated analyses of reference zircon standards  
189 Mud Tank and SA01. The obtained <sup>176</sup>Hf/<sup>177</sup>Hf value on the two reference materials are identical  
190 to the reported values within uncertainties, 0.282504 ± 0.000042 (2s, n=31) for Mud Tank zircon  
191 and 0.282285 ± 0.000047 (2s, n=15) for SA01 zircon<sup>27,35</sup>.

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