A scanning ion imaging investigation into the micron-scale U-Pb systematics in a complex lunar zircon

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Abstract

The full U-Pb isotopic systematics in a complex lunar zircon ‘Pomegranate’ from lunar impact breccia 73235 have been investigated by the development of a novel Secondary Ion Mass Spectrometry (SIMS) scanning ion imaging (SII) technique. This technique offers at least a four-fold increase in analytical spatial resolution over traditional SIMS analyses in zircon. Results from this study confirm the hypothesis that the Pomegranate zircon crystallized at 4.302 ± 0.013 Ga and experienced an impact that formed, U-enriched zircon around primary zircon cores at 4.184 ± 0.007 Ga (2σ , all uncertainties). The increase in spatial resolution offered by this technique has facilitated targeting of primary zircon that was previously inaccessible to conventional spot analyses. This approach has yielded results indicating that individual grains with a diffusive distance of less than ~ 4 μm have been reset to the young impact age, while individual grains with a diffusive distance larger than ~ 6 μm have retained the old crystallization age. Assuming a broad range in cooling rate of 0.5-50 $^{\circ}\text{C}/\text{year}$, which has been observed in a suite of similar lunar breccias, a maximum localized temperature generated by the impact that reset small primary zircon and created new, high-U zircon is estimated to be between 1100 and 1280 $^{\circ}\text{C}$.

Introduction

Zircon (ZrSiO_4) is one of the most robust and widely used geochronometers, in large part owing to its exceptional physical and chemical stability. It also has the advantage of hosting three independent decay chains to Pb, two of which have a common parent element in U. The robust physical characteristics of zircon have the potential to preserve geochemical and chronological information through multiple

40 overprinting events postdating the original crystallization. However, this ability to
41 preserve multiple event results in complexities often observed in the U-Pb system
42 within individual zircon grains. As a result, the complex U-Pb isotopic information of
43 some individual zircon grains has been interpreted to represent partial recrystallization,
44 replacement, and variable damage of crystal lattice by radioactive decay. Recent
45 studies (e.g., Grange et al., 2013a; Kusiak et al., 2013a,b; Moser et al., 2011;
46 Whitehouse et al., 2014) indicate that this variability and mixing can occur on the
47 scale of a few nanometers to several micrometers. Consequently, the correct
48 interpretation of U-Pb data requires understanding of processes that might affect the
49 chemistry and/or internal structure of zircon on an appropriate scale. When applied to
50 zircon chronology in the samples from the Moon, where the fundamental objective is
51 to constrain both igneous activity and the subsequent impact history of the lunar
52 surface, the analytical challenge is to determine ages of recognized impact features
53 that are often less than a few micrometers in the smallest dimension and may have
54 irregular shapes (e.g., Grange et al., 2009; Grange et al., 2011; Grange et al., 2013a,b;
55 Timms et al., 2012).

56

57 The small size and irregular shape of impact features in lunar zircon make them
58 difficult, if not impossible to recognize and analyze for U-Pb systematics by
59 conventional *in situ* methods, such as spot analysis using Secondary Ion Mass
60 Spectrometry (SIMS) even at small spot sizes (e.g., Liu et al., 2011). Our aim in this
61 manuscript is to present data obtained using a newly developed and tested SIMS
62 scanning ion-imaging (SII) approach on the previously described “Pomegranate”
63 zircon from lunar breccia 73235 (Grange et al., 2013b; Pidgeon et al., 2007; Smith et
64 al., 1986). In contrast to earlier studies that measured only Pb-isotopes by SII, we

65 have developed analytical and data processing protocols that resolve full U-Pb
66 systematics in irregularly shaped regions of interest with minimum dimension of <5
67 μm .

68

69 ***Sample details:***

70 Sample 73235 is an impact melt breccia with several mineral and lithic clasts
71 (25%) surrounded by an aphanitic matrix (75%) that has been previously described in
72 great detail (Pidgeon et al., 2007; Ryder 1993; Spudis and Ryder, 1981). The crew of
73 Apollo 17 collected this sample from landslide material associated with the South
74 Massif of the Taurus-Littrow Valley on the edge of the Serenitatis impact basin (Wolf,
75 1981). Due to the large range of shock features (e.g., shocked anorthosites and
76 feldspathic impactites), rounded edges on mineral clasts, and bulk chemistry with a
77 KREEP affinity, this sample has been interpreted as being part of the Serenitatis
78 ejecta sequence that has incorporated both local and exotic materials (Dence et al.,
79 1976; Hodges and Kushiro, 1974; Rhodes et al., 1974; Ryder, 1993; Spudis and Ryder,
80 1981; Wolf, 1981). Thin section 82 of sample 73235 contains a rounded clast of 75
81 broken fragments of zircon that resembles a cluster of pomegranate seeds (hence, the
82 name commonly applied to this feature) located in a patch of 25 plagioclase crystals
83 that have reacted with the surrounding matrix (Pidgeon et al., 2007; Smith et al.,
84 1986). The bulk sample has an $^{40}\text{Ar}/^{39}\text{Ar}$ age of $3.90 \pm 0.04/0.08$ Ga and a Rb-Sr
85 whole rock isochron age of 4.02 ± 0.01 Ga, which has been hypothesized to be the
86 age of breccia formation (Nyquist et al., 1974; Ryder, 1993).

87

88 ***Pomegranate Zircon:***

89 The Pomegranate zircon aggregate has been investigated extensively by
90 backscatter electron (BSE) imaging, secondary electron (SE) imaging,
91 cathodoluminescence (CL), and electron backscatter diffraction mapping (EBSD), and
92 its U-Pb isotopic systematics were studied by *in situ* SIMS U-Pb analyses utilizing a
93 Köhler illuminated primary spot of ~20 µm in diameter (Grange et al., 2013; Pidgeon
94 et al., 2007; Smith et al., 1986). The CL patterns of the larger grains within the
95 aggregate have different but uniform CL density and retain primary, igneous sector
96 zoning (Figure 1). However, these features are not traceable across fragments, which
97 implies that the grains have been displaced and rotated during impact (Figure 1; after
98 Pidgeon et al., 2007). Therefore, this zircon has been interpreted to have formed as a
99 single large grain in an anorthosite, which subsequently experienced an impact or
100 impacts, resulting in fragmentation (Pidgeon et al., 2007; Smith et al., 1986).
101 Surrounding primary igneous zircon there is secondary zircon that is low in surface
102 relief, dark in CL, non-crystalline (i.e., not indexed in EBSD, Figure 1 and Figure 2;
103 Grange et al., 2013; Pidgeon et al., 2007).

104

105 Traditional spot U-Pb analyses by SIMS indicate that the large primary grains
106 and secondary zircon have $^{207}\text{Pb}/^{206}\text{Pb}$ ages of 4.315 ± 0.015 Ga and 4.187 ± 0.011 Ga,
107 respectively (uncertainties are 2σ , Pidgeon et al., 2007). U-Pb ages for this zircon do
108 not overlap the previously determined Ar-Ar (3.9 Ga) or the Rb-Sr ages of the bulk
109 sample (4.02 Ga; Nyquist et al., 1974; Ryder, 1993), which indicates that the thermal
110 pulse responsible for these ages had no significant effect on either the primary or
111 secondary zircon. The U concentration in the secondary zircon (86-215 µg/g) is
112 significantly higher than that of the primary zircon (generally 14-106 µg/g, Pidgeon et
113 al., 2007). The current interpretation on the formation of this zircon, taking into

114 account the morphology of the zircon aggregate and U-Pb ages of fragments and
115 high-U zircon, is that the fragments date the initial crystallization of zircon and its
116 anorthosite host, whereas the amorphous matrix was likely to have been formed by
117 cataclasis and frictional heating during the impact-induced shock that resulted in the
118 loss of all radiogenic Pb and addition of U, thereby locally resetting the zircon U-Pb
119 system (Pidgeon et al., 2007). However, the source of the additional U remains
120 unexplained. During this impact event, the primary igneous zircon was fragmented,
121 rotated, displaced and engulfed by the newly formed, high-U impact-generated zircon.
122

123 The proposed impact resulted in a heterogeneous assemblage of relatively
124 large primary CL active grains, small primary CL active grains, and secondary high-U
125 zircon. The large primary grains and the areas of secondary zircon, which are large
126 enough for traditional SIMS analyses, were targeted in previous studies. However, the
127 majority of the secondary zircon shows irregular distribution between the fragments,
128 with individual patches that are generally smaller than accessible by conventional *in*
129 *situ* SIMS U-Pb spot analyses. Similarly, the small primary, CL active grains are
130 smaller than a conventional SIMS spot. In the present study we use the new SII
131 approach to image five areas that cover both primary and secondary zircon (Figure 1),
132 allowing for the investigation of the U-Pb systems at high spatial resolution (<5 μm)
133 in this highly complex zircon aggregate.

134

135 **Analytical Methods**

136 Zircon standards were analyzed in 25 mm diameter polished epoxy mounts
137 and the Pomegranate zircon in lunar sample 73235 was analyzed in a 25 mm diameter,
138 standard (30 μm thickness) petrographic thin section of the lunar breccia. A Tescan

139 MIRA3 FE-SEM at the Microscopy and Microanalysis Facility (John de Laeter
140 Centre) at Curtin University in Perth, Western Australia, was used to image the
141 Pomegranate grain with CL and BSE at an acceleration voltage of 12 kV. Prior to
142 scanning ion image acquisition, the lunar thin section was cleaned in alternating
143 ultrasonic baths of ethanol and distilled water. After the sample was cleaned it was
144 coated with 30 nm of Au. Similar procedures were followed for the epoxy mount
145 containing standard zircon grains.

146 All SIMS SII analyses were performed using a CAMECA IMS1280 large-geometry
147 ion microprobe located at the NordSIMS facility, Swedish Museum of Natural
148 History, Stockholm, Sweden, closely following protocols modified from those
149 described by Kusiak et al. (2013a, b) and Whitehouse et al. (2014). Prior to the start of
150 each SII analysis, an area of 80 x 80 μm was sputtered for 10 minutes using a $\sim 20 \mu\text{m}$,
151 $\sim 10 \text{ nA O}_2^-$ primary beam to effectively remove surface gold coating and minor
152 residual surface contamination. Additionally, this approach resulted in a uniform
153 implantation of oxygen in the upper surface of the zircon over the image area,
154 enhancing secondary ion yield. To obtain high spatial resolution for the 70 x 70 μm
155 ion images of the standards and unknowns, a 50 μm primary beam aperture was used,
156 resulting in a projected (Köhler) beam diameter of $\sim 5 \mu\text{m}$ on the sample surface, a
157 beam current of $\sim 250 \text{ pA}$. This spot size, which corresponds to a surface area of 20
158 μm^2 , defines the minimum spatial resolution at which the technique can confidently
159 resolve individual, non-mixed zircon chemical domains.

160 The secondary ion beam from the rastered area was processed using the dynamic
161 transfer optical system (DTOS), a synchronized raster in the transfer section of the
162 instrument that deflects the ions back onto the ion optic axis of the instrument

163 regardless of their original point of sputtering from the sample, allowing both image
164 acquisition and collection of secondary ions at high mass resolution, in this case
165 $M/\Delta M$ of 4860. Secondary ion intensities of ^{204}Pb , ^{206}Pb , ^{207}Pb , and ^{208}Pb were
166 measured simultaneously using the multi-collector array, while ions of $^{90}\text{Zr}^{16}\text{O}$, ^{238}U
167 and $^{238}\text{U}^{16}\text{O}_2$ were measured in subsequent peak hops. As with conventional SIMS U-
168 Pb in zircon analyses on the IMS1270/80 instruments, oxygen was introduced into the
169 sample chamber increasing its pressure to $\sim 2 \times 10^{-5}$ mbar, effectively doubling Pb
170 sensitivity (Schuhmacher et al., 1993). All ion-counting detectors used in this study
171 were low-noise discrete dynode Hamamatsu 416 electron multipliers; typical
172 backgrounds of <0.005 cps obviating the need for any background correction.

173 Prior to image acquisition, the secondary ion beam was centered in the field aperture,
174 optimized for maximum energy distribution in a 45 eV energy window, and mass
175 calibrated using the zircon matrix $^{90}\text{Zr}^{16}\text{O}$ signal. Count times for the ions of $^{90}\text{Zr}^{16}\text{O}$,
176 simultaneous Pb isotopes, ^{238}U , and $^{238}\text{U}^{16}\text{O}_2$ were 2, 12, 5, and 2 seconds,
177 respectively, and images were integrated over 60 scans, resulting in a total analysis
178 time of 34 minutes. $^{90}\text{Zr}^{16}\text{O}$, ^{238}U , and $^{238}\text{U}^{16}\text{O}_2$ ions were measured in the same
179 detector as the ^{208}Pb ions in order to minimize aging on detectors used for the critical
180 radiogenic Pb isotopes, ^{206}Pb and ^{207}Pb , as well as unradiogenic ^{204}Pb .

181 Images were processed using the CAMECA WinImage2 software package. Regions
182 of interest (ROIs) were selected in each of the five Pomegranate images. ROIs were
183 selected based on the 'hot spots' in ^{206}Pb images for each Pomegranate image (Figure
184 3). These hotspots correspond to the impact-generated zircon that have dark CL and
185 are low relief in SE images, and have very little crystallinity (Figure 2). Additionally,
186 several ROIs were selected in the primary zircon. The same ROI outlines were then

187 applied to two images of the 91500 zircon standard and one image of an external
188 zircon standard, Temora 2. Once the ROIs were determined for each image, the
189 isotope ratios of $^{207}\text{Pb}/^{206}\text{Pb}$, $^{208}\text{Pb}/^{206}\text{Pb}$, $^{204}\text{Pb}/^{206}\text{Pb}$, $^{206}\text{Pb}/^{238}\text{U}$, $^{238}\text{U}^{16}\text{O}_2/^{238}\text{U}$, and
190 $^{90}\text{Zr}_2^{16}\text{O}/^{238}\text{U}$ were calculated in WinImage2 and exported to Microsoft Excel where
191 all isotope ratios were corrected for gain differences between the electron multipliers
192 and U/Pb elemental fractionation as described below.

193 A single, linear correction factor that accounts for Pb isotope mass
194 fractionation and gain differences between electron multipliers was determined by
195 performing conventional spot analyses of USGS basaltic glass reference material
196 BCR-2G (~11 $\mu\text{g/g}$ Pb) before the imaging session, following the methods described
197 by Bellucci et al. (2015) and Whitehouse et al. (2005), using an ~20 μm , ~10 nA O_2^-
198 primary beam. All spot analyses were conducted in multi-collector mode at a mass
199 resolution of 4860 ($M/\Delta M$), using an NMR field sensor in regulation mode to control
200 the stability of the magnetic field. Gain correction values were then determined by
201 comparison to the accepted Pb isotope ratios for BCR-2G (Woodhead and Hergt,
202 2000). Since Pb isotope fractionation is effectively non-existent between different
203 materials in SIMS Pb isotope analysis, typically being within the level of individual
204 analytical uncertainties (Shimizu and Hart, 1982), this correction is all that is needed
205 to account for gain/mass fractionation differences in Pb isotopic ratios during multi-
206 collector SIMS analyses. External precision in this session for ratios $^{207}\text{Pb}/^{206}\text{Pb}$,
207 $^{208}\text{Pb}/^{206}\text{Pb}$, and $^{204}\text{Pb}/^{206}\text{Pb}$ was 0.5%, 0.4%, and 0.6%, respectively (2σ). This
208 external precision was propagated through to all unknown Pb isotopic ratios.

209 Determining the correct $^{238}\text{U}/^{206}\text{Pb}$ ratio from the secondary ion elemental-
210 ratio ($^{238}\text{U}^+/^{206}\text{Pb}^+$) is the most important factor for accurate U-Pb age determinations

211 (e.g., Jeon and Whitehouse, 2015). In conventional spot analyses, factors that affect
212 this ratio are a result of the combination of variations in sample chamber pressure,
213 sample surface(s) irregularities, and changing primary beam conditions
214 (shape/intensity). Since the goal for this study is to determine the spatially resolved U-
215 Pb systematics, an examination of how the U/Pb ratio varies across the image is
216 critical.

217 The simplest way of correcting for U/Pb fractionation uses the $^{206}\text{Pb}/^{238}\text{U}^{16}\text{O}_2$
218 ratio, since UO_2 has the most similar energy distribution in a secondary ion beam to
219 Pb (Jeon and Whitehouse, 2015; Stern 2000; Stern and Amelin, 2003). The
220 $^{206}\text{Pb}/^{238}\text{U}^{16}\text{O}_2$ was determined for each ROI in each image, after correcting for gain
221 differences between the detector used for ^{208}Pb and ^{206}Pb , a ROI-specific correction
222 factor to convert $^{206}\text{Pb}/^{238}\text{U}^{16}\text{O}_2$ to $^{206}\text{Pb}/^{238}\text{U}$ was calculated by averaging two
223 different analyses of zircon 91500 using the same ROI, assuming an accepted age of
224 1065 Ma (Wiedenbeck et al., 1995). This correction factor was then applied to each
225 specific unknown ROI, compensating for any U/Pb fractionation differences across
226 the imaged area. The accuracy of the fractionation factor determined from 91500 was
227 assessed by applying the fractionation factor for each ROI to an image of the
228 secondary zircon standard, Temora 2 (Figure 4). The uncertainty (std. dev.) in the
229 average between the two 91500 ROIs was propagated in quadrature through to the
230 final, corrected U/Pb ratios.

231 In summary, the final, corrected isotopic ratios are a result of an external gain
232 and external U/Pb correction and the reported uncertainty thus combines Poisson's
233 counting statistics (internal uncertainty), the external gain, and the external U/Pb
234 correction. Lastly, common Pb (given in Table 1 as $f_{206}\%$), which likely results

235 primarily from sample preparation, was monitored using ^{204}Pb and corrected
236 assuming the present-day terrestrial Pb composition of Stacey and Kramers (1975).
237 After the gain, U/Pb fractionation, and common Pb corrections were made for each
238 ROI, its U and Pb concentrations, $^{207}\text{Pb}/^{206}\text{Pb}$ and $^{206}\text{Pb}/^{238}\text{U}$ ages were calculated
239 (ages assuming the U decay constant recommendation of Steiger and Jäger, 1977),
240 and weighted average calculations and concordia diagrams were made with Isoplot
241 4.15 (Ludwig, 2012).

242

243 **Results**

244 Results for all corrected ratios, common Pb correction (resulting from any
245 residual laboratory contamination), U concentrations and calculated Th/U ratios (from
246 measured $^{208}\text{Pb}/^{206}\text{Pb}$ and age) for each image and corresponding ROIs for Temora 2
247 and Pomegranate (Figure 3) are presented in Table 1. Results from the measured
248 $^{206}\text{Pb}/^{238}\text{U}^{16}\text{O}_2$ for the two sequential 91500 images (blue and red bars), concordia
249 diagrams, and weighted $^{206}\text{Pb}/^{238}\text{U}$ ages for Temora 2 are shown in Figure 4. All of
250 the individual calculated ages for Temora 2 are within uncertainty of the accepted
251 $^{206}\text{Pb}/^{238}\text{U}$ age of 416.8 ± 1.3 Ma (Black et al., 2004), but are systematically older than
252 the accepted value and have variable levels of discordancy, which will be discussed in
253 the following section. Concordia diagrams for all Pomegranate ROIs are presented in
254 Figure 5; similar to the Temora 2 analyses, some analyses are discordant outside of
255 uncertainty and speculation on the origins of this discordancy will be addressed in the
256 discussion section.

257

258 **Discussion**

259 ***U-Pb correction***

260 The measured $^{206}\text{Pb}/^{238}\text{U}^{16}\text{O}_2$ ratio of all ROIs in two ion images of 91500 is
 261 similar (~ 0.15) but shows some dependence on location of each ROI in the image
 262 (Figure 3 and 4). Most notably, the right-hand side of all of images has a lower
 263 $^{206}\text{Pb}/^{238}\text{U}^{16}\text{O}_2$ ratio than the center/left sides of the images (e.g., Image 3 ROI-2). The
 264 decrease in $^{206}\text{Pb}/^{238}\text{U}^{16}\text{O}_2$ ratio is likely due to the incoming primary beam traversing
 265 over areas where the gold coating has been removed. Specifically, since the primary
 266 beam enters from the left of the image, it travels a greater distance over a non-gold
 267 coated surface to reach the right side than the left side of the image. A similar Pb/U
 268 bias effect has been observed when spot analyses are performed to the right of
 269 previous analyses. Despite these small differences in $^{206}\text{Pb}/^{238}\text{U}^{16}\text{O}_2$, using the average
 270 $^{206}\text{Pb}/^{238}\text{U}^{16}\text{O}_2$ ratio for each ROI from the calibration standard, then applying that
 271 correction to the unknown, identical ROI, defines a set of statistically
 272 indistinguishable $^{206}\text{Pb}/^{238}\text{U}$ ages in Temora 2 regardless of location on the image.
 273 When combined into a weighted mean, these data yield robust statistics for each
 274 image (i.e., $\text{MSWD} < 1$, $P > 0.75$) indicating a statistically identical population.
 275 However, for each image the $^{206}\text{Pb}/^{238}\text{U}$ age in Temora 2 is slightly older, while
 276 within error, of the accepted age of 417 Ma, with a maximum of difference of $\sim 5\%$
 277 seen in the weighted averages of the ROIs from image 5 (Figure 4).

278 ***Concordance, accuracy, precision, and potential limitations***

279 The calculated $^{206}\text{Pb}/^{238}\text{U}$ ages for Temora 2 measured here are systematically
 280 older by a small amount. Despite the systematic bias, all individual Temora 2 analyses
 281 are within 2σ uncertainty of concordia and the accepted age and for the weighted
 282 average for each image (Figure 4). However, if all Temora 2 ROIs are pooled it
 283 results in a $^{206}\text{Pb}/^{238}\text{U}$ age of 429 ± 7 Ma (2σ), which is outside of uncertainty of the

284 accepted age of 416.8 ± 1.3 Ma. Given the systematic offset, pooling of $^{206}\text{Pb}/^{238}\text{U}$
285 ages is not recommended using the method described here. This limits the potential
286 application of a single ROI U-Pb analyses because in small areas the precision can be
287 significantly worse than a traditional SIMS analysis, depending on size of the area and
288 counting statistics.

289 The origin of the systematic offset could be a result of a small error in the
290 actual $^{206}\text{Pb}/^{238}\text{U}$ age of 91500 for the grains used for calibration. Isotope dilution
291 TIMS analyses of 91500 indicate that some pieces of 91500 are discordant
292 corresponding to 0.5 % Pb loss (Wiedenbeck et al., 1995). Another potential
293 explanation for discordance is that there could be relief differences between standard
294 mounts and grains embedded in thin sections. However, the likeliest possibility is
295 having too few standard 91500 images when compared to conventional U-Pb in
296 zircon analyses. During traditional U-Pb in zircon analyses, there are at least 6-8 U-Pb
297 calibration standards, often many more. Having only two standards reduces the ability
298 to recognize and filter out a potential outlier standard. As such, any future U-Pb ion
299 imaging studies need to have significantly more primary and ideally secondary
300 standards to ensure accuracy in pooled $^{206}\text{Pb}/^{238}\text{U}$ ages. However, for this work, the
301 small offset in $^{206}\text{Pb}/^{238}\text{U}$ is not significant, because it does not affect ages calculated
302 from $^{207}\text{Pb}/^{206}\text{Pb}$ ratios, which are used to access chronology in old (including lunar)
303 zircon because of the improved accuracy.

304

305 ***U-Pb systematics and ages in Pomegranate zircon***

306 Regardless of textural context, statistically pooling all $^{207}\text{Pb}/^{206}\text{Pb}$ ages into the
307 oldest and youngest statistically equal but independent populations (weighted

308 averages of $MSWD < 1.5$ and $P > 0.05$) yields ages of 4.302 ± 0.013 (2σ , $MSWD =$
309 1.3 , $P = 0.16$) and 4.184 ± 0.007 Ga (2σ , $MSWD = 0.71$, $P = 0.84$, Figure 6). These
310 two statistically distinct $^{207}\text{Pb}/^{206}\text{Pb}$ age groups are in excellent agreement with the
311 4.315 ± 0.015 and 4.187 ± 0.011 Ga ages determined by Pidgeon et al. (2007) and
312 Smith et al. (1986). The older age group encompasses large, CL-active grains, while
313 the younger group contains small, CL-active grains and high-U, secondary zircon.
314 This is a notable result given the measurements were performed using three different
315 instruments using two different techniques: spot analysis SHRIMP I and SHRIMP II,
316 and SII analysis on a CAMECA IMS1280 using the technique described here.

317 The secondary zircon clearly is enriched in both U and Pb and has a decreased
318 calculated Th/U when compared to the primary host zircon (Table 1, Figures 2, 3, 7),
319 which is also in agreement with previous observations by Pidgeon et al. (2007) and
320 Smith et al. (1986). This zircon areas is clearly located on the crystal boundaries,
321 associated with dark areas in CL, and yield very low band contrast in EBSD patterns
322 indicating very little crystallinity, which supports the hypothesis that this high-U, low
323 Th/U (now amorphous) zircon was formed during an impact at 4.187 Ga.

324 Here the smallest ROI defined from the U and Pb enriched area (Image 5,
325 ROI-6) is $\sim 8 \mu\text{m}^2$, which corresponds to an improvement of 40x in spatial resolution
326 over a $20 \mu\text{m}$ spot typical in conventional SIMS analysis ($314 \mu\text{m}^2$). However, this
327 particular analysis is smaller than the spot size used ($20 \mu\text{m}^2$) and likely represents a
328 mixed Pb signal, though dominated by the targeted concentration ‘hotspot’.
329 Additionally, the largest area here defined in Image 4, ROI-2 is $800 \mu\text{m}^2$, which is 2.5
330 times larger than a traditional spot. This advantage over traditional spot analyses
331 could be widely applicable to homogenous, low Pb areas to increase counting

332 statistics.

333 In addition to the two previously identified types of zircons (Grange et al.,
334 2013; Pidgeon et al., 2007; and Smith et al., 1986), a third type is now recognized that
335 have low U concentrations and elevated Th/U ratios, yet have been completely reset
336 to the younger age (Figure 7). Importantly, this third group is comprised of both the
337 secondary, high-U zircon (i.e., Image 1, ROIs-5, -6, -7, -8, -9, Figure 3) and CL active
338 primary zircon (Image 2, ROIs-1, -2, -3, -4, Image 5, ROI-1, Figure 3). The previous
339 hypothesis of impact generation very likely applies to the young zircon in Image 1
340 (Figure 3), due to their location on the rims of the primary zircon and correlation with
341 low relief, lack of CL, and featureless EBSD patterns. This cannot, however, explain
342 the CL active, reset primary zircon (in Images 2 and 5, Figure 3). The young ages
343 observed in these ROIs can only result from resetting of the U-Pb system due to
344 diffusion during the impact that formed the secondary zircon.

345 ROIs-1, -2, -3, and -4 in Image 2 (Fig. 2) and ROI-1 in Image 5 all have low U
346 abundances (12-65 $\mu\text{g/g}$), high calculated Th/U (0.35-0.6, Table 1), are active in CL,
347 are surrounded by high U zircon, and are therefore assumed to be primary zircon,
348 despite being reset to the young 4.187 Ga age (Figures 3 and 4). For Image 2, the
349 ROIs-1, -2, -3, -4, are very small with area dimensions of 77, 49, 76, and 29 μm^2 ,
350 respectively, and are completely reset. Conversely, Image 3 ROI-4, which is similarly
351 CL active but has the dimensions of 173 μm^2 , was not reset. The smallest effective
352 diffusive distance (from the middle of the grain to the rim) in the reset primary grains
353 is $\sim 4 \mu\text{m}$ and the smallest effective diffusive distance in the non-reset primary grain is
354 $\sim 6 \mu\text{m}$.

355 The closure temperature of a mineral – that is, the temperature at the time

corresponding to its apparent age – is determined by grain size, diffusion coefficients, mineral geometric factor, and cooling rate (e.g., Dodson et al., 1973). Therefore, the localized temperature experienced by the impact-reset grains can be estimated making the following assumptions: Pb diffusion coefficients in zircon (Cherniak and Watson, 2001), a spherical geometry factor, and a range in cooling rate of 0.5-50°C/year (determined in multiple lunar impact breccias by Cushing et al., 1999). Figure 8 illustrates the effective diffusion radius vs. closure temperature in zircon based on the range in cooling rates determined for other lunar breccias. The temperature range that is capable of completely resetting the grains with a minimum effective diffusion radius smaller than 4 µm without significantly modifying grains larger than 6 µm is between ~1100 and 1280 °C (Figure 8). It is noteworthy that larger grains do not show any measurable diffusive resetting and the boundary between impact generated zircon and large primary zircon is extremely sharp (i.e., Image 3 ROI-3 and ROI-4). This is most likely due to localized temperature/pressure variability associated with the impact event that generated the impact zircon.

Conclusions

The new, extended scanning ion imaging technique facilitates a ~4x increase in spatial resolution for full U-Pb systematics, while being capable of accommodating analysis domains of any user-defined shape and accessing the visual distribution of U and Pb which is in contrast to full U-Pb spot analyses, even when performed at 5 µm. Application of this technique has allowed for the determination of accurate $^{206}\text{Pb}/^{238}\text{U}$ age in Temora 2 zircon (within an individual ROI, at the 2σ uncertainty level), and the $^{207}\text{Pb}/^{206}\text{Pb}$ ages of three texturally and compositionally distinct types of domain corresponding to primary, reset primary, and amorphous, impact generated zircon in the Pomegranate aggregate from lunar sample 73235. Combined with previous data,

381 the Pomegranate grains crystallized at 4.302 ± 0.013 Ga and experienced an impact
382 event that generated new, high-U zircon and reset primary zircons with a minimum
383 diffusive distance of $<6 \mu\text{m}$ at 4.184 ± 0.007 Ga (errors 2σ). Assuming that localized
384 resetting of the U-Pb system occurred by volume diffusion during a thermal pulse
385 associated with an impact event, a comparison of the extent of $^{207}\text{Pb}/^{206}\text{Pb}$ age
386 resetting with grain size (effective diffusion distance) in the primary zircon vs.
387 preserved primary zircon constrains the maximum localized temperature generated by
388 the impact to between 1100 and 1280 °C. Diffusion modeling at this spatial resolution
389 would not have been possible using *in situ* SIMS conventional spot analysis
390 techniques. Lastly, this study demonstrates that this method could be widely
391 applicable to understanding isotope systematics in complex zircon as long as the
392 limitations discussed in this manuscript are taken into account. Given that this is
393 currently the only analytical technique that can resolve the spatial distribution of U,
394 Pb, and the U-Pb isotope systematics on the scale of $<5 \mu\text{m}$ simultaneously, the
395 approach and future improvements outlined in this study could significantly enhance
396 what can be learned from complex zircon.

397

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408

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527

528 **Figure 1.** Backscatter Electron (BSE) and cathodoluminescence (CL) images of
529 Pomegranate zircon in lunar impact breccia 73235, previously described by Pidgeon
530 et al., (2007). Areas of ion imaging analyses are marked by red squares that are 70 x

531 70 μm . Bright spots in the BSE image are remnants of the Au coating used during
532 SIMS analyses.

533

534 **Figure 2.** $^{90}\text{Zr}_2^{16}\text{O}$ and ^{206}Pb ion images, EBSD and Secondary Electron (SE) images,
535 of primary igneous (Zr 1) and secondary high-U zircon (Zr 2), corresponding to
536 Image 1 of Figure 1. Zr 2 is clearly enriched in ^{206}Pb compared to Zr 1, despite its
537 younger age. SE and EBSD images from Grange et al. (2013).

538

539 **Figure 3.** Ion images of $^{90}\text{Zr}_2^{16}\text{O}$, ^{238}U , ^{206}Pb , CL, and selected regions of interest
540 (ROIs) superimposed on ^{206}Pb ion image.

541

542 **Figure 4.** Measured $^{206}\text{Pb}/^{238}\text{U}^{16}\text{O}_2$ in two 91500 images (ROIs are given by number
543 above blue and red bars with values for first and second images of 91500,
544 respectively), corrected Temora 2 concordia diagrams, and weighted average of
545 $^{206}\text{Pb}/^{238}\text{U}$ age in Temora 2. Green dashed line in weighted average calculation is the
546 accepted value for the $^{206}\text{Pb}/^{238}\text{U}$ age of Temora 2 (417 Ma, Black et al., 2004).

547

548 **Figure 5.** Concordia diagrams for all ROIs in Pomegranate zircon. Red ellipses are
549 the statistically younger population, while black ellipses are the statistically older
550 population.

551

552 **Figure 6.** $^{207}\text{Pb}/^{206}\text{Pb}$ age weighted averages for the primary zircon (A) and reset
553 zircon (B). Dashed green lines are values determined by traditional U-Pb analyses by
554 Pidgeon et al. (2007) and Smith et al. (1986).

555

556 **Figure 7.** U concentration in $\mu\text{g/g}$ and calculated Th/U vs. $^{207}\text{Pb}/^{206}\text{Pb}$ age. Grey field
557 represents previously determined values by traditional *in situ* methods from Pidgeon
558 et al. (2007) and Smith et al. (1986). Zircon 1 field is dominated by old, CL active
559 grains. Zircon 2 field is dominated by young, CL inactive, high-U grains. Zircon 3
560 field is a combination of low U, CL active/inactive, and young ages.

561

562 **Figure 8.** Effective diffusive distance in micrometers vs. closure temperature in $^{\circ}\text{C}$
563 determined by the method of Dodson (1973). Closure T was calculated using Pb
564 diffusion coefficients (Cherniak and Watson, 2001), assuming a spherical geometry,
565 and a range in closure temperatures observed in other lunar breccias (Cushing et al.,
566 1999).

Figure 2

Zircon orientation

~70 μm

Figure 3

Figure 5

Figure 6

Figure 8

Sample	Image #	Area of Interest (ROI)	Area Analyzed in m ²	[U in g/g]	Th/U calc ^a	²⁰⁶ Pb/ ²⁰⁸ Pb	1 %	2σRSD	²⁰⁷ Pb/ ²⁰⁸ Pb	1 %	²⁰⁶ Pb/ ²⁰⁸ Pb	1 %	²⁰⁶ Pb/ ²⁰⁸ Pb	1 %	Disc. % conv.	Disc. % 2	1m	²⁰⁷ Pb/ ²⁰⁸ Pb	1 %	²⁰⁶ Pb/ ²⁰⁸ Pb	1 %	²⁰⁷ Pb/ ²⁰⁸ Pb	1 %	²⁰⁶ Pb/ ²⁰⁸ Pb Age (Ma)	1	²⁰⁷ Pb/ ²⁰⁸ Pb Age (Ma)	1	CL Active	Group
Pomegranate	1	1	272	6	0.81	0.00116	34	2.2	80	6.5	1.12	5.9	0.96	29	22	3	0.515	1.9	0.129	3.7	4845	204	4284	27	Yes	Old			
		2	224	6	0.85	0.00108	35	2.0	82	6.5	1.11	6.1	0.96	22	5	5	0.516	2.0	0.122	3.7	4937	216	4287	29	Yes	Old			
		3	288	23	0.87	0.00059	28	1.1	77	4.2	1.08	4.1	0.96	15	4	4	0.506	1.9	0.134	2.1	4776	138	4287	17	Yes	Old			
		4	159	6	0.84	0.00010	50	0.3	83	9.2	1.11	8.9	0.95	18	1	1	0.506	2.2	0.135	4.9	4866	308	4287	12	Yes	Old			
		5	139	5	0.83	0.00010	50	0.3	83	9.2	1.11	8.9	0.95	18	1	1	0.506	2.2	0.135	4.9	4866	308	4287	12	Yes	Old			
		6	114	54	0.83	0.00012	71	0.2	77	9.6	1.16	9.5	0.99	27	3	3	0.480	1.4	0.044	3.4	4876	338	4181	20	No	Young			
		7	114	0.20	0.00005	100	0.1	66	9.4	1.00	9.3	0.99	9.3	0.99	37	6	0.479	1.3	0.046	3.4	4460	306	4178	19	No	Young			
		8	18	46	0.83	0.00073	58	1.4	79	11	1.23	10	0.96	37	6	6	0.464	2.8	0.061	6.6	5165	374	4132	41	No	Young			
		9	87	61	0.23	0.00054	33	1.0	65	6.7	0.99	6.6	0.98	9	4	4	0.475	1.4	0.053	3.5	4422	214	4165	20	No	Young			
Pomegranate	2	1	44	18	0.84	0.00034	100	0.4	74	8.8	1.12	8.4	0.95	23	9	9	0.482	2.7	0.090	5.4	4847	391	4186	23	Yes	Young			
		2	49	26	0.85	0.00000	0	0.0	64	12	0.97	12	0.96	15	6.8	48	0.478	3.8	0.082	6.8	4864	381	4175	48	Yes	Young			
		3	76	61	0.69	0.00184	26	3.4	66	8.9	1.02	8.7	0.97	13	13	3.6	0.468	2.0	0.110	3.6	4532	280	4145	29	Yes	Young			
		4	29	65	0.47	0.00033	100	0.6	65	11	0.97	11	0.96	6	0.485	3.2	0.485	1.6	0.033	5.7	4377	332	4197	46	Yes	Young			
		5	102	0.31	0.00005	71	0.1	74	4.6	1.10	1.10	4.5	0.98	20	2	9	0.485	0.9	0.065	2.1	4785	154	4195	14	No	Young			
		6	74	118	0.26	0.00019	58	0.4	63	5.3	0.94	5.2	0.96	2	0.489	1.4	0.489	0.6	0.064	3.3	4265	163	4210	21	No	Young			
		7	139	12	0.27	0.00010	12	0.0	63	7.2	1.05	7.0	0.98	18	1.2	1.2	0.485	1.2	0.056	4.9	4871	346	4185	29	No	Young			
		8	23	137	0.23	0.00000	0	0.0	63	11	0.95	11	0.98	4	0.481	2.0	0.481	0.6	0.056	4.9	4812	366	4185	29	No	Young			
		9	23	137	0.23	0.00000	0	0.0	63	11	0.95	11	0.98	4	0.481	2.0	0.481	0.6	0.056	4.9	4812	366	4185	29	No	Young			
Pomegranate	3	1	533	29	0.89	0.00144	14	2.7	78	8.7	1.08	9.3	0.97	14	15	15	0.524	0.9	0.196	1.3	4735	297	4311	14	Yes	Old			
		2	243	23	0.75	0.00124	27	2.3	72	9.5	1.03	8.7	0.98	9	0.524	1.6	0.524	0.6	0.171	2.5	4557	312	4271	24	Yes	Old			
		3	301	29	0.69	0.00060	33	1.3	72	4.7	1.01	4.5	0.95	6	0.516	1.5	0.516	0.6	0.161	2.4	4448	197	4289	24	Yes	Old			
		4	172	28	0.68	0.00016	58	0.3	72	9.2	1.05	9.2	0.98	2	0.489	1.4	0.489	0.6	0.034	2.8	4807	135	4186	21	Yes	Old			
		5	159	18	0.83	0.00016	28	0.3	71	3.1	1.04	3.7	0.98	18	0.485	0.7	0.485	0.3	0.034	2.8	4807	135	4186	21	Yes	Old			
		6	91	130	0.26	0.00033	33	0.6	72	4.1	1.05	3.9	0.96	6	0.485	1.1	0.485	0.6	0.056	2.6	4622	131	4197	16	No	Young			
		7	166	130	0.27	0.00067	20	1.2	64	3.9	0.97	3.8	0.97	6	0.481	0.9	0.481	0.9	0.065	2.1	4377	121	4183	14	No	Young			
		8	762	12	0.93	0.00311	12	5.8	84	11	1.16	11	0.99	21	1.6	1.6	0.525	1.1	0.192	1.6	4953	389	4314	17	Yes	Old			
		9	800	9	0.75	0.00096	19	1.9	81	11	1.10	11	0.99	15	0.533	1.5	0.533	1.5	0.162	2.3	4785	397	4337	21	Yes	Old			
Pomegranate	4	1	134	11	0.83	0.00016	19	0.3	81	6.6	1.10	8.4	0.99	15	0.533	1.5	0.533	1.5	0.162	2.3	4785	397	4337	21	Yes	Old			
		2	154	11	0.83	0.00016	19	0.3	81	6.6	1.10	8.4	0.99	15	0.533	1.5	0.533	1.5	0.162	2.3	4785	397	4337	21	Yes	Old			
		3	276	132	0.16	0.00071	71	1.3	69	6.2	1.04	6.0	0.97	14	0.482	1.4	0.482	1.4	0.066	2.3	4694	201	4186	21	No	Young			
		4	505	20	0.47	0.00090	35	1.7	84	12	1.15	12	0.97	21	0.527	3.2	0.527	3.2	0.088	6.4	4945	435	4318	47	Yes	Old			
		5	64	117	0.30	0.00026	50	0.5	84	4.8	1.15	4.7	0.98	15	0.480	1.0	0.480	1.0	0.086	4.4	4615	156	4181	15	No	Young			
Pomegranate	5	1	222	12	0.60	0.00146	38	2.7	73	9.0	1.09	8.6	0.98	18	2.9	2.9	0.488	2.5	0.127	4.3	4741	297	4204	37	Yes	Young			
		2	134	12	0.60	0.00146	38	2.7	73	9.0	1.09	8.6	0.98	18	2.9	2.9	0.488	2.5	0.127	4.3	4741	297	4204	37	Yes	Young			
		3	154	11	0.77	0.00017	29	1.4	72	8.8	1.13	14	1.00	30	0.517	0.8	0.517	0.8	0.147	1.1	5178	510	4202	12	Yes	Old			
		4	505	37	0.64	0.00074	29	1.4	72	14	0.97	14	1.00	30	0.536	1.4	0.536	1.4	0.158	2.2	4381	459	4343	10	Yes	Old			
		5	118	143	0.21	0.00012	50	0.2	74	3.8	1.13	3.7	0.97	25	0.472	1.0	0.472	1.0	0.043	2.8	4875	128	4155	15	No	Young			
		6	8	161	0.47	0.00000	0	0.0	72	18	1.12	18	0.96	24	0.467	4.8	0.467	4.8	0.095	9.2	4836	638	4139	69	No	Young			
		7	71	163	0.21	0.00026	45	0.5	68	12	1.05	12	0.99	3.5	0.475	1.3	0.475	1.3	0.064	3.5	4614	421	4164	19	No	Young			
		8	154	153	0.23	0.00016	38	2.7	73	9.0	1.09	8.6	0.98	18	2.9	2.9	0.488	2.5	0.127	4.3	4741	297	4204	37	Yes	Young			
		9	30	97	0.27	0.00018	100	0.3	75	7.7	1.14	7.4	0.95	25	0.479	2.4	0.479	2.4	0.055	5.9	4892	258	4177	34	No	Young			
Temora 2	1	1	272	90	0.82	0.00000	0	0.0	80	6.8	1.09	3.3	0.48	-29	0.060	6.0	0.060	0.139	4.1	430	14	598	124	Yes	Young				
		2	224	82	0.82	0.00000	0	0.0	80	6.8	1.09	3.3	0.48	-29	0.060	6.0	0.060	0.139	4.1	430	14	598	124	Yes	Young				
		3	288	86	0.82	0.00000	0	0.0	80	6.8	1.09	3.3	0.48	-29	0.060	6.0	0.060	0.139	4.1	430	14	598	124	Yes	Young				
		4	159	86	0.82	0.00000	0	0.0	80	6.8	1.09	3.3	0.48	-29	0.060	6.0	0.060	0.139	4.1	430	14	598	124	Yes	Young				
		5	139	87	0.82	0.00000	0	0.0	80	6.8	1.09	3.3	0.48	-29	0.060	6.0	0.060	0.139	4.1	430	14	598	124	Yes	Young				
		6	114	63	0.82	0.00000	0	0.0	80	6.8	1.09	3.3	0.48	-29	0.060	6.0	0.060	0.139	4.1	430	14	598	124	Yes	Young				
		7	71	63	0.82	0.00000	0	0.0	80	6.8	1.09	3.3	0.48	-29	0.060	6.0	0.060	0.139	4.1	430	14	598	124	Yes	Young				
		8	18	72	0.82	0.00000	0	0.0	80	6.8	1.09	3.3	0.48	-29	0.060	6.0	0.060	0.139	4.1	430	14	598	124	Yes	Young				
		9	87	72	0.82	0.00000	0	0.0	80	6.8	1.09	3.3	0.48	-29	0.060	6.0	0.060	0.139	4.1	430	14	598	124	Yes	Young				
Temora 2	2	1	77	167	0.82	0.00000	0	0.0	80	6.8	1.09	3.3	0.48	-29	0.060	6.0	0.060	0.139	4.1	430	14	598	124	Yes	Young				
		2	49	159	0.82	0.00000	0	0.0	80	6.8	1.09	3.3	0.48	-29	0.060	6.0	0.060	0.139	4.1	430	14	598	124	Yes	Young				
		3	123	123	0.82	0.00000	0	0.0	80	6.8	1.09	3.3	0																