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Every zircon deserves a date: selection bias in detrital geochronology

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Abstract

Detrital zircon geochronology can help address stratigraphic- to lithospheric-scale geological questions. The approach is reliant on statistically robust, representative age distributions that fingerprint source areas. However, there is a range of biases that may influence any detrital age signature. Despite being a fundamental and controllable source of bias, handpicking of zircon grains has received surprisingly little attention. Here, we show statistically significant differences in age distributions between bulk-mounted and handpicked fractions from an unconsolidated heavy mineral sand deposit. Although there is no significant size difference between bulk-mounted and handpicked grains, there are significant differences in their aspect ratio, circularity and colour, which indicate inadvertent preferential visual selection of euhedral and coloured zircon grains. Grain colour comparisons between dated and bulk zircon fractions help quantify bias. Bulk-mounting is the preferred method to avoid human-induced selection bias in detrital zircon geochronology.

1. Introduction

Detrital zircon U–Pb geochronology is a powerful tool in deciphering Earth's sedimentary archive, able to answer a myriad of research questions including: sediment transfer (e.g. Luo *et al.* 2014); maximum depositional ages (e.g. Nelson, 2001); tectonomagmatic processes (e.g. Wotzlaw *et al.* 2011); palaeogeographic correlations (e.g. Samson *et al.* 2005); or crustal evolution (e.g. Amelin *et al.* 1999). According to Fedo *et al.* (2003), we can distinguish between two strategies in detrital zircon geochronology: (i) qualitative analysis that strives for representation of every age mode within the detrital record, regardless of their relative abundance (e.g. Gehrels & Ross, 1998); and (ii) quantitative analysis that endeavours to obtain representative age distributions (e.g. Li *et al.* 2019), or a combination of both strategies (e.g. McWilliams *et al.* 2010). Although sound reasons exist to carry out qualitative analysis, the advent of high-*n* acquisition techniques (e.g. Pullen *et al.* 2014) and readily available statistical tools (e.g. Sircombe & Hazelton, 2004) have certainly provoked a shift towards quantitative analysis as the preferred approach during the last 10–15 years, allowing for quantifiable similarities among different geological domains. Quantifying relationships between samples makes use of the relative abundance of age modes (e.g. Nie *et al.* 2018), which is often facilitated using statistical assessment to maintain objectivity (e.g. Vermeesch, 2013).

The underlying assumption for a geologically meaningful interpretation of inter-sample comparison of detrital zircon age distributions is that the analysed samples are a true reflection of the sediment sampled and that this can be used as a proxy for the relative proportion of crystalline rocks in the source region. However, this foundational assumption may be undermined by a number of biases that can be simplified to those associated with (i) geological processes, and (ii) methodological approaches (Chew *et al.* 2020; Fig. 1). Intrinsic biases are inherent to geological processes, for instance variations in mineral fertility (e.g. Moecher & Samson, 2006), variable erosion rates (e.g. Spencer *et al.* 2017), sedimentary sorting effects (e.g. Lawrence *et al.* 2011) or selective upgrading, such as removal of metamict grains during transport (e.g. Markwitz & Kirkland, 2018). Several studies have highlighted the necessity to quantify methodological limitations in detrital zircon geochronology datasets to allow robust interpretations (e.g. Ibañez-Mejia *et al.* 2018). Methodological biases can be divided into analytical biases and biases induced during sample processing. While substantial efforts have been made to establish a common practice for analytical procedures (e.g. Garzanti *et al.* 2018) and data processing for *in situ* U–Pb analysis (Košler *et al.* 2013; Horstwood *et al.* 2016; Spencer *et al.* 2016), less agreement exists in workflows and equipment used for zircon separation between laboratories. Mineral processing procedures have significant potential for introducing systematic biases (Sláma & Košler, 2012; Chew *et al.* 2020). Any systematic bias that alters the

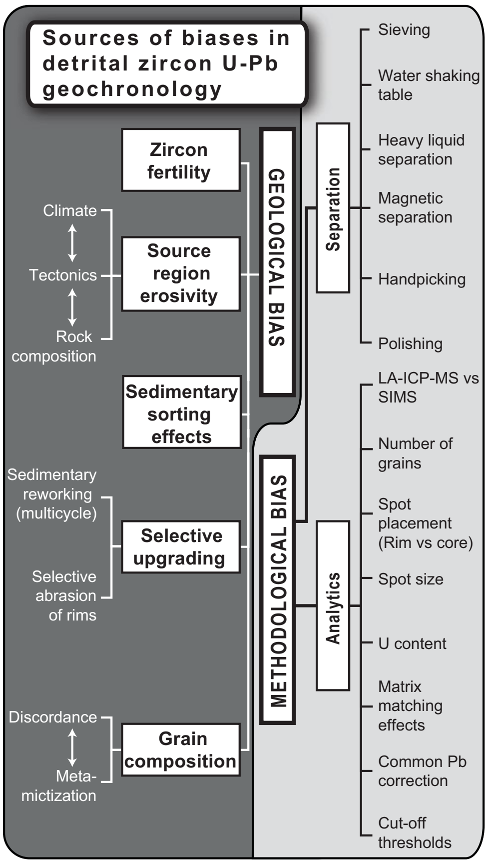

Fig. 1. Simplified overview of sources of bias in detrital zircon geochronology. Biases may be intrinsic to laboratory procedures (methodological bias) or may be a function of the zircon material itself and its geological environment (geological bias).

true proportions of age modes inevitably enhances the risk of erroneous interpretation based on statistical inter-sample comparison.

Chew *et al.* (2020) reviewed biases in single-grain provenance analysis and concluded that preferential operator selection of certain mineral populations was responsible for selective bias during handpicking. However, mineral handpicking, conceivably the part of zircon separation most likely affected by human-induced selection bias, has not received much consideration in the literature. Although automated mineral mapping may reduce the need for handpicking (e.g. Hrstka *et al.* 2018; Lünsdorf *et al.* 2019), handpicking remains the most-used mounting technique applied in detrital zircon geochronology (Gaudette *et al.* 1981; Sláma & Košler, 2012; online Supplementary Fig. S1, available at http://journals.cambridge.org/geo). Handpicking may have some benefits in certain fields of geochronology, for example where more pristine volcanic crystals may lead to more precise crystallization age determination, and improve maximum depositional age constraint. Nonetheless, because zircon shape and colour is known to vary with geological history and composition (e.g. Markwitz & Kirkland, 2018), different age modes will likely be characterized by different grain characteristics that could influence their selection in any detrital zircon study. This study provides the first evidence of preferential selection bias (based on grain shape and colour) induced during handpicking in a natural sample. Consequently, this work highlights a significant methodological pitfall concerning the use of relative age peaks in detrital zircon U–Pb geochronology.

2. Geological setting

The Scott Coastal Plain (Fig. 2) represents a suitable area to evaluate selection bias in detrital zircon. The area has a well-understood crystalline basement with distinct age modes, grain shape and colour variability (Makuluni *et al.* 2019). The plain is a piedmont alluvial surface comprising strandlines with heavy mineral sand deposits of economic significance (Baxter, 1977). The succession of siliciclastic coastal sediments unconformably overlies Palaeozoic and Mesozoic strata of the Perth Basin, is bordered by the Neoproterozoic–Palaeozoic Pinjarra Orogen and the Proterozoic Albany–Fraser Orogen, and overlies the Archean Yilgarn Craton (Baddock, 1995).

The South West Terrane of the Archean Yilgarn Craton contains Meso-Neoarchean zircons with a predominant age mode at *c.* 2700–2600 Ma (Mole *et al.* 2019). The Albany–Fraser Orogen reflects the Proterozoic modification of the Yilgarn Craton and records key tectonomagmatic events at *c.* 1710–1650, *c.* 1345–1260 and *c.* 1215–1140 Ma (Kirkland *et al.* 2011). The Leeuwin Complex is one of the few inliers of the Pinjarra Orogen and comprises age modes at *c.* 1100–1000, *c.* 750 and *c.* 520 Ma (Collins, 2003; Fitzsimons, 2003).

3. Methods

This study was motivated by the observation of a significant discrepancy between age spectra of bulk-mounted and handpicked subsamples (the two most commonly employed grain-mounting techniques; online Supplementary Fig. S1) of heavy mineral concentrates from the unconsolidated Governor Broome heavy mineral sand deposit (34° 15' 21" S, 115° 24' 24" E). In this work, no experimental design to test selection bias existed *a priori*, that is, age data were primarily acquired during conventional zircon U–Pb geochronology sessions for the purpose of sedimentary provenance analysis. The 53–1000 μm grain size fraction underwent separation using a liquid with a density of 2.96 g cm^{-3} and isodynamic magnetic separation resulting in a zircon-dominated mineral separate permitting selection of the widest possible range of grain characteristics while picking zircon grains. Handpicking was performed using a stereo binocular microscope and needle, attempting representativeness. A representative split (coning and quartering) of the heavy mineral concentrate (bulk-mounted) and handpicked grains were affixed in the same resin mount, enabling consistency for image analysis.

Detrital zircon U–Pb geochronology was performed using laser ablation – inductively coupled plasma – mass spectrometry (LA-ICP-MS) at Curtin University's John de Laeter Centre (Perth, Australia). Full details of the sample preparation and U–Pb geochronology procedure are provided in online Supplementary Materials S1 and S2 (including Tables S1 and S2) (available at http://journals.cambridge.org/geo).

Grain shape and colour analyses were performed for concordant measurements (i.e. measurements intercepting Concordia) on transmitted light images (online Supplementary Material S3) acquired after geochronological measurements using an automated Zeiss AXIO Imager M2m microscope system. Grain shape analysis was conducted using ImageJ (Abramoff *et al.* 2004). To facilitate colour comparison of the two populations, the numbers of colours were simplified to 14 RGB values determined by the ImageJ plugin 'Color Inspector 3D' (using histogram mode). Frequencies of these indexed colours were calculated for individual grains using a Python script. To assess relative colour difference

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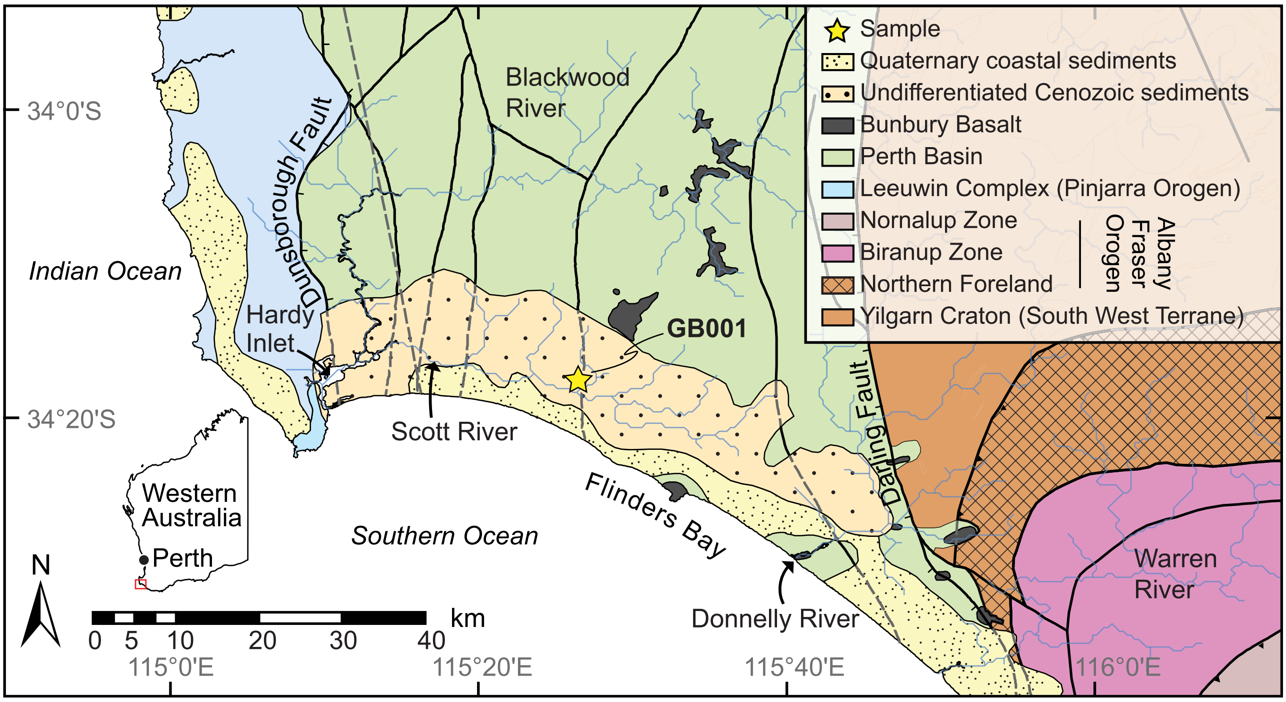

Fig. 2. (Colour online) Geological map of the Scott Coastal Plain in Western Australia. Red rectangle on inset indicates study area. GB001 indicates the Governor Broome heavy mineral sand deposit used in this study.

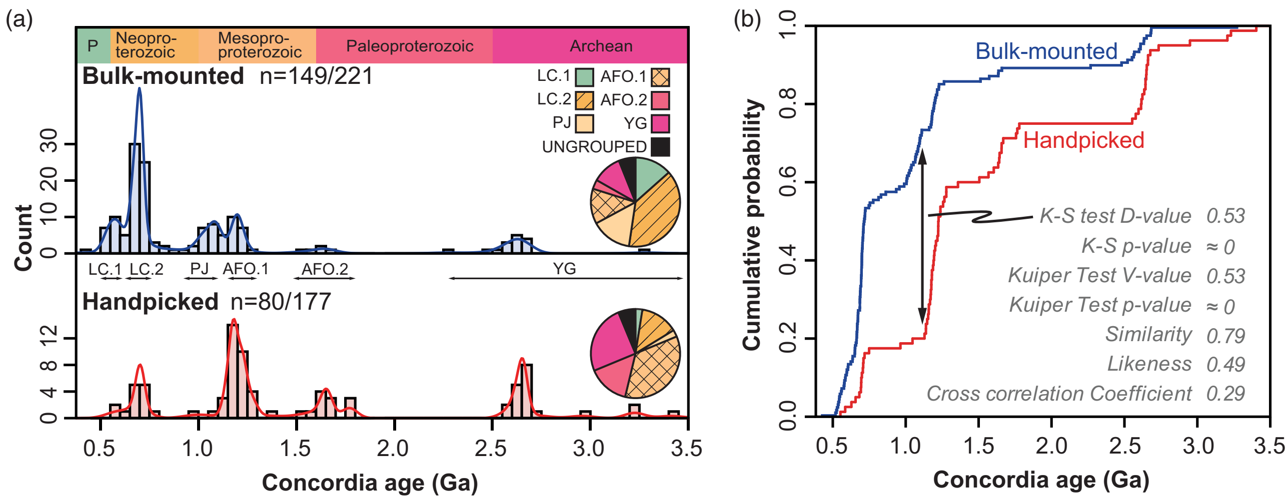

Fig. 3. (Colour online) Results of detrital zircon U–Pb geochronology. (a) Normalized kernel density estimates for bulk-mounted and handpicked populations. Arrows show different age modes used in this study, and the pie charts visualize the fraction of age modes in the two populations. P – Phanerozoic. (b) Cumulative age distributions and metrics to diagnose similarity in inter-sample comparison.

between grains, we use the sum of these colour frequencies while omitting rim artefacts (R40, G40, B40; approximately dark-grey) and background resin transmission (R128, G128, B128; approximately grey), labelled 'Σ Colour' [%].

4. Results

A total of 229 concordant detrital zircon ages range from the Palaeoarchean to early Phanerozoic. Bulk-mounted (*n*, concordant analyses/all analyses = 149/221) and handpicked (*n* = 80/177) populations show polymodal age distributions with varying intensities of major age modes at *c.* 630–510 Ma (% bulk-mounted/% handpicked = 13/3), *c.* 760–640 Ma (39/14), *c.* 1110–900 Ma (15/3), *c.* 1300–1150 Ma (13/35), *c.* 1800–1500 Ma (3/15) and *c.* 2750–2500 Ma (11/25) (Fig. 3a, b; online Supplementary Material S2, Table S3).

The two populations show no significant difference in grain areas (Fig. 4a; online Supplementary Material S2, Table S4); individual bulk-mounted grains range from 5706 to 18 605 µm^2 (mean ± standard deviation, 9540 ± 2389 µm^2) compared with 4983 to 25 435 µm^2 (9295 ± 2815 µm^2) for handpicked grains. In contrast, the aspect ratio shows significant differences between

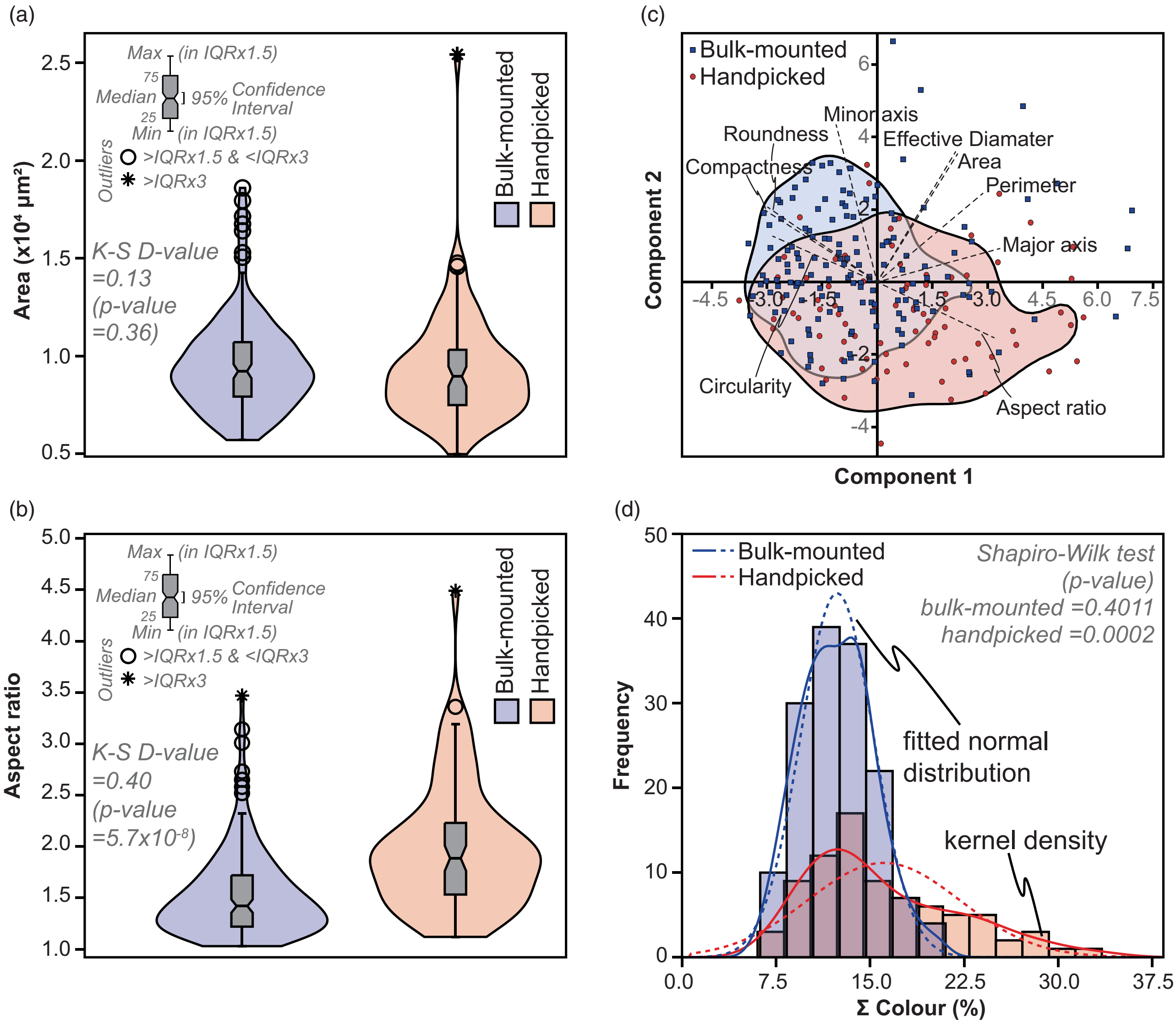

Fig. 4. (Colour online) Results of grain characteristics. (a) Area (IQR – interquartile range) and (b) aspect ratio of bulk-mounted (blue) and handpicked (red) populations. (c) Principal component analysis of bulk-mounted and handpicked populations. Principal components 1 and 2 have variance values of 62.37% and 32.88%, respectively. (d) Colour histogram of bulk-mounted (blue) and handpicked (red) populations.

the two populations ranging from 1.03 to 3.47 (1.54 ± 0.43) for bulk-mounted grains compared with 1.12 to 4.49 (1.97 ± 0.60) for handpicked grains (Fig. 4b). Similarly, grain circularity (4π ×area/perimeter^2; 1 = a perfect circle) indicates distinguishable populations. Bulk-mounted grains range from 0.52 to 0.89 (0.74 ± 0.07) compared with 0.38 to 0.86 (0.70 ± 0.09) for handpicked grains. Principal component analysis (clustering visualization among multivariate data) based on grain shape parameters shows bulk-mounted and handpicked populations form partial overlapping clusters and indicate aspect ratio is a primary characteristic defining differences between the populations (Fig. 4c).

The value of Σ Colour (high values = colourful grains) of bulk-mounted grains ranges from 6.02 to 20.45% (12.36 ± 2.92%) compared with 7.50 to 33.49% for handpicked grains (16.08 ± 6.05%, Fig. 4d; online Supplementary Material S2, Table S5). The handpicked population features higher skewness of Σ Colour than the bulk-mounted population (0.85 compared with 0.26). The Shapiro–Wilk test for normality rejects the null hypothesis that the handpicked population is normally distributed (P-value < 0.05).

5. Discussion

5.a. Provenance

To facilitate comparison, detrital zircon ages are grouped into naturally occurring age modes in the study area (online Supplementary Material S2, Table S6). Age modes 630–510, 760–640 and 1100–900 Ma can be linked to the proximal Leeuwin Complex (Pinjarra Orogen). Ages within the intervals 1300–1150 and 1800–1500 Ma are most likely derived from the Albany–Fraser Orogen, and ages of 2750–2500 Ma are likely sourced from the Yilgarn Craton. All age modes can therefore be readily correlated to proximal crystalline sources and their former east Gondwana equivalents, in accordance with regional sedimentary rocks (i.e. Perth Basin) that show similar original

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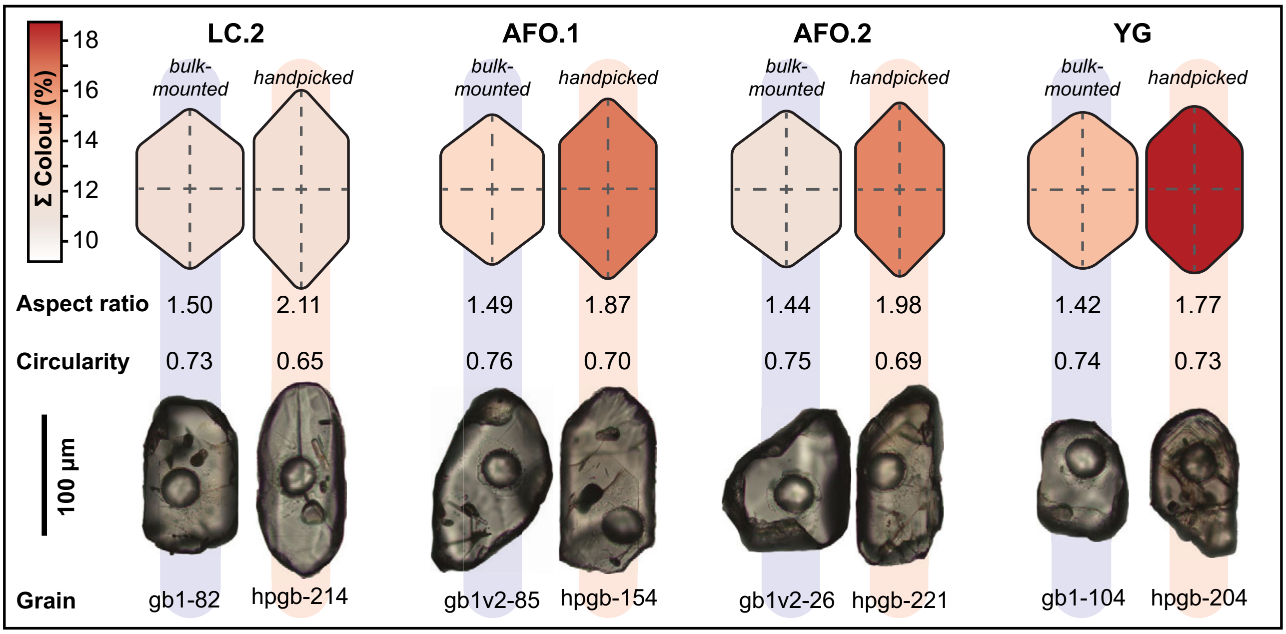

Fig. 5. (Colour online) Comparison of bulk-mounted and handpicked populations for the four major age modes. Upper row shows schematic drawings of the average grain based on length of major and minor axis, circularity and Σ Colour. Lower row displays transmitted light images of representative zircon grains based on average aspect ratio and Σ Colour of their population.

source areas (Olierook *et al.* 2019). Consequently, we interpret the sediment was derived from reworking of local sediments and primary basement erosion.

5.b. Handpicking-induced sampling bias

The qualitative interpretation of sedimentary provenance (i.e. identification of source regions) is only marginally different between bulk-mounted and handpicked populations (similarity coefficient of *c.* 0.79). However, commonly used population comparison metrics that are more sensitive to the relative abundance of age modes (Kolmogorov–Smirnov and Kuiper tests) suggest the bulk-mounted and handpicked subsamples are statistically distinguishable, which contradicts their true relationship (Fig. 3b). Our data suggest handpicking of zircon separates can produce a biased (i.e. non-random) zircon population. Any subsequent statistical evaluation comparing the handpicked population to a reference age distribution may lose geological meaning.

We correlate the sampling bias of age modes to preferential selection of more euhedral and more colourful zircon grains (Fig. 5). In contrast to the size-control picking bias for synthetic zircon populations proposed by Sláma & Košler (2012), we cannot resolve significant differences between the grain size (here, area) of bulk-mounted and handpicked zircons for our natural sample. However, results of artificially rounded (air-abrasion) zircons displaying ‘spherical or near-spherical shape’ (Sláma & Košler, 2012) cannot be readily compared with the natural counterpart used in this work as the latter are expected to exhibit more natural complexity (e.g. primary crystal morphologies). The use of synthetic samples is therefore likely incapable of fully unravelling controls of potential handpicking bias as it can diminish naturally existing sources of bias during handpicking. Non-random sampling during handpicking of a natural sample is most prominent in the aspect ratio among grain shape parameters. The median aspect ratio of the handpicked population intersects the bulk-mounted population above its 75th percentile (Fig. 4a), and aspect ratio accounts for substantial variance between bulk-mounted and handpicked populations based on principal component analysis. We therefore interpret a significant preference for euhedral (high aspect ratio) grain shapes. Similarly, differences in the colour frequencies, for example, non-normal distribution of the handpicked population (skewed towards more colourful zircons) compared with the normally distributed bulk-mounted population, and the significant higher abundance of coloured grains in the handpicked subsample, are interpreted as preferential selection of coloured grains (Fig. 4d). These interpretations are consistent with visual object recognition models stressing the role of shape (Biederman, 1987) as well as colour (Bramão *et al.* 2011). Zircon grains showing features of higher visibility or stereotypical appearance (e.g. euhedral grain surfaces or colour) are more readily perceived, and can therefore become overrepresented during operator selection.

Variations in grain characteristics are correlated with changes in the proportion of age modes. In this study, the oldest age mode grains (AFO.1, AFO.2 and YG) are overrepresented in the handpicked subsample. Overrepresented age mode grains in the handpicked fraction show higher aspect ratios (mean of 1.95) and a higher proportion of coloured grains (*c.* 17%) relative to the bulk-mounted subsample, while the younger age mode LC.2 lacks significant colour difference from its bulk-mounted equivalent (*c.* 12%) and is underrepresented (Fig. 6). While grain shapes can become extensively modified during transport, grain colour remains more faithful to its origin. The ability to quantify the dissimilarity in colour between bulk-mounted and handpicked zircon fractions therefore provides a means to constrain the magnitude of selection bias and potentially address its influence on the detrital zircon age fingerprint. We used the colour difference between the bulk-mounted zircon population and handpicked zircon grains to measure bias derived from preferential selection of coloured grains (Fig. 7). Adjusting the proportions of age modes

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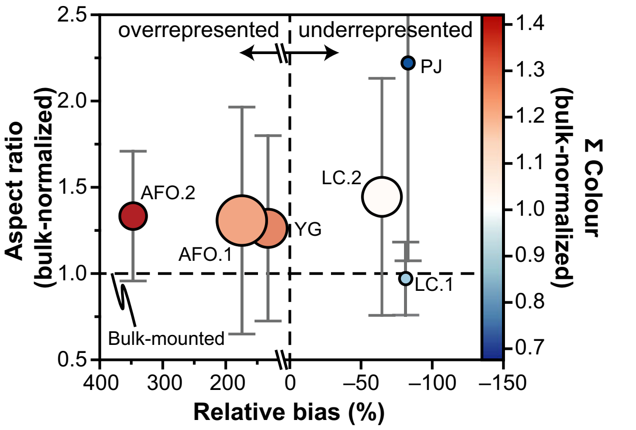

Fig. 6. (Colour online) Comparison of bulk-mounted-normalized aspect ratio and colour for major age modes. Relative bias indicates relative offset to bulk-mounted population and is calculated as {[(fraction of age mode handpicked) − (fraction of age mode bulk-mounted)]/(fraction of age mode bulk-mounted)} × 100. The bubble size scales with fraction of age mode in the handpicked population. Error bars are 1 standard deviation.

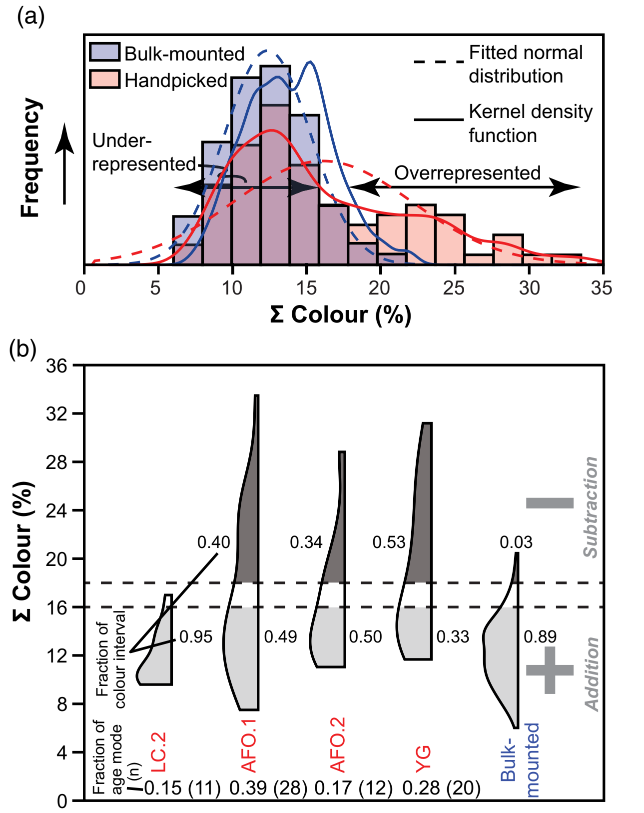

Fig. 7. (Colour online) Assessing the degree of bias. (a) Colour histogram of bulk-mounted (blue) and handpicked (red) populations used to calculate the adjustment of age modes. (b) Σ Colour distribution for major age modes of the handpicked population. For details and calculation see Discussion and online Supplementary Materials S1 and S2 (Table S7).

in the handpicked sample according to the calculated colour bias improved overall similarity (calculation in online Supplementary Material S1; see also online Supplementary Material S2, Table S7). Nonetheless, persistence of significant differences compared with the bulk-mounted age distribution remains and is attributed to a complex interplay of grain characteristics controlling grain selection, as well as non-unique age-mode grain colour relationships. Regardless, capturing the relative colour differences between bulk-mounted and measured handpicked zircons can identify the presence and magnitude of bias.

The results presented in this study cast doubt on the often assumed randomness of handpicked age distributions used for inter-sample comparison. Although handpicking may be a preferred approach when targeting specific populations (e.g. to constrain the maximum depositional age) or to capture every age mode of the detrital record, studies interested in representative age distributions should whenever possible avoid handpicking. Individual studies and those referring to them, as well as studies making use of the global detrital record, will be positively impacted in terms of statistical robustness by omitting a potential source of bias. Increasing numbers of publications relating to global compilations of detrital zircon as a tracer of the Earth's crustal dynamics (e.g. Reimink *et al.* 2021) are inevitably incorporating original bias into their interpretation, potentially impairing this powerful approach. These findings also demonstrate that documenting grain-mounting techniques is imperative for reliable inter-sample comparison.

Selection bias in detrital zircon geochronology is an ultimate function of variability among zircon shapes and colours, as well as zircon concentration of the mineral separate. Lower concentration of zircons and more uniform characteristics might reduce the chance of inducing sampling bias during handpicking. Analytical conditions of this work are consistent with the vast majority of published detrital zircon studies, that is, a single hand-picking operator and small to medium sample size (n) of analysed zircons. Following the calculation of Vermeesch (2004), 149 and 80 (concordant) grains means no fraction of the population comprising more than 0.041 and 0.068, respectively, is missed at the 95% confidence level. The examined number of grains is therefore sufficient to characterize the zircon cargo of the parent population.

We therefore conclude that these results are a valid reflection of possible bias relevant to most detrital zircon studies interested in statistical inter-sample comparison. Consequently, we argue that the use of automated mineral mapping to target analysis of bulk-mounted grains offers important advantages in decreasing sample-treatment-induced bias and improves the robustness of detrital zircon data.

6. Conclusions

Handpicking of detrital zircon from a natural sample produced a statistically different age distribution compared with bulk-mounting of the same material. This bias would considerably impact subsequent statistical evaluation if unrecognized. The significant variation in grain shape and colour suggests the preferential manual selection of euhedral and coloured grains. An assessment of the discrepancy between bulk-mounted and handpicked zircons can therefore be used to evaluate the degree of representativeness of handpicked grains. These results highlight the importance of minimizing sample handling steps whenever practicable. Zircon bulk-mounting is the preferred approach for detrital zircon geochronology studies reliant on representative age distributions.

Supplementary material. To view supplementary material for this article, please visit https://doi.org/10.1017/S0016756821000145

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Conflict of interest. None.

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