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U-Pb detrital zircon dates and provenance data from the Beaufort Group (Karoo Supergroup) reflect sedimentary recycling and air-fall tuff deposition in the Permo-Triassic Karoo foreland basin

Pia A. Viglietti [a, *], Dirk Frei [b], Bruce S. Rubidge [a], Roger M.H. Smith [a, c]

[a] Evolutionary Studies Institute, School for Geosciences, University of the Witwatersrand, Johannesburg, Private Bag 3 Wits, 2050, South Africa
[b] Department of Earth Sciences, University of the Western Cape, Private Bag X17, 7530 Bellville, South Africa
[c] Iziko South African Museum of Cape Town, P.O. Box 61, Cape Town, 8000 South Africa

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ABSTRACT

Detrital zircon U-Pb age dating was used for provenance determination and maximum age of deposition for the Upper Permian (upper Teekloof and Balfour formations) and Lower Triassic (Katberg Formation) lithostratigraphic subdivisions of the Beaufort Group of South Africa's Karoo Basin. Ten samples were analysed using laser ablation - single collector - magnetic sectorfield - inductively coupled plasma - mass spectrometry (LA-SF-ICP-MS). The results reveal a dominant Late Carboniferous-Late Permian population (250 ± 5 Ma – 339 ± 5 Ma), a secondary Cambrian-Neoproterozoic (489 ± 5 Ma to 878 ± 24 Ma) population, a minor Mesoproterozoic (908 ± 24 Ma to 1308 ± 23) population, and minor occurrences of Devonian, Ordovician, Proterozoic and Archean zircon grains. Multiple lines of evidence (e.g. roundness and fragmentary nature of zircons, palaeo-current directions, and previous work), suggest the older zircon populations are related to sedimentary recycling in the Gondwanide Orogeny. The youngest and dominant population contain elongate euhedral grains interpreted to be directly derived from their protolith. Since zircons form in felsic igneous rocks, and no igneous rocks of Late Permian age occur in the Karoo Basin, these findings suggest significant input of volcanic material by ash falls. These results support sedimentological and palaeontological data for a Lopingian (Late Permian) age for the upper Beaufort Group, but contradict previous workers who retrieved Early Triassic dates from zircons in ashes for the Beaufort and Ecca Groups. Pb-loss not revealed by resolvable discordance on the concordia diagram, and metamictization of natural zircons are not factored into the conclusions of earlier workers.

1. Introduction

The zirconium silicate, zircon ($ZrSiO_4$), forms in a wide variety of contrasting rock types such as acid to intermediate igneous, volcanic, and metamorphic rocks. Exhibiting high refractory behaviour and mechanical resistance under sedimentary conditions, zircons represent reliable closed systems that are important for obtaining accurate and precise U-Th-Pb ages concerning source rock petrology and chronology (Fedo et al., 2003). Also, the radiometric age obtained for the youngest zircon grain within a population of detrital zircons is considered to be a good indicator of the maximum age for deposition of the sedimentary (different from an absolute age), or can indicate the timing of metamorphism responsible for affecting sedimentary rocks after deposition (Zeh et al., 2008; Andersen, 2013; Vorster, 2013; Orejana et al., 2015; Andersen et al., 2016). This makes zircons ideal mineral tracers in sedimentary systems, recording potential tectonic and sedimentary processes active in the geological past (von Eynatten and Dunkl, 2012).

Laser ablation - single collector - magnetic sectorfield - inductively coupled plasma - mass spectrometry (LA-SF-ICP-MS) and sensitive high-resolution ion microprobe reverse geometry (SHRIMP-RG) have been previously conducted on the rocks of the Karoo Supergroup to discern provenance and also maximum ages of deposition (Fildani et al., 2007, 2009; Bowden, 2013; Vorster, 2013; McKay et al., 2015). LA-SF-ICP-MS method is the more versatile and economic technique for geochronological age

* Corresponding author.
E-mail addresses: pia.viglietti@gmail.com (P.A. Viglietti), dfrei@uwc.ac.za (D. Frei), bruce.rubidge@wits.ac.za (B.S. Rubidge), rsmith@iziko.org.za (R.M.H. Smith).

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determinations of not only zircons, but is also useful in dating other minerals such as Titanite, Phlogopite, and Apatite. This study investigates changes in provenance of Upper Permian and Lower Triassic sedimentary rocks of South Africa's Karoo Basin by employing LA-SF-ICP-MS dating of detrital zircons. Lithological units which correlate to this time interval include the Balfour Formation, occurring in the central and south eastern part of the basin, the Upper Teekloof Formation in the southwest, and the Lower Triassic Katberg Formation in the central basin (Fig. 1). The youngest ages in the samples were also used as a rough guide for maximum age of deposition for each lithological unit. In addition, a petrographic study was conducted to determine whether these results corroborated detrital zircon data.

2. Materials and methods

Detrital zircon dating and petrographic descriptions were undertaken to assist with provenance investigations of the Upper Permian Karoo Basin lithostratigraphic units to determine whether changes in provenance could be documented through the Upper Permian and Lower Triassic strata. Sandstone-rich lithostratigraphic units were chosen for sampling as the densest minerals and largest particles accumulate at the bases of channel deposits (Fedo et al., 2003; Vorster, 2013). A total of ten samples were collected for detrital zircon dating, and 11 samples were collected for petrographic description. These samples were collected from the lower Balfour Formation (Sample 1), upper Teekloof Formation (Samples 2 and 3), upper Balfour Formation (Samples 4, 5, 6, 7), and lower Katberg Formation (Samples 8, 9, and 10) (Fig. 1).

2.1. Detrital zircon analysis

Detrital zircon U-Pb age dating was performed at the Central Analytical Facility (CAF), Stellenbosch University. Zircons were separated from the samples using standard mineral separation techniques (crushing, sieving, panning, heavy liquid separation, magnetic barrier separation, picking to optical clearness under a binocular microscope). Zircons were embedded in standard 1-inch round epoxy mounts, ground to reveal zircon cross-sections and polished for examination of internal structures by CL imaging. All U–Pb age data obtained was acquired by laser ablation - single collector - magnetic sectorfield – inductively-coupled plasma mass-spectrometry (LA-SF-ICP-MS) employing a Thermo Finnigan Element 2 mass spectrometer coupled to a Resonetics Resolution S155 excimer laser ablation system (Jackson et al., 2004). All age data presented here were obtained by single spot analyses with a spot diameter of 30 μm and a crater depth of approximately 15–20 μm. The methods employed for analysis and data processing are described in detail by Frei and Gerdes (2009) and Cornell et al. (2016). For quality control, the 91500 (Wiedenbeck et al., 1995), Plesovice (Sláma et al., 2008) and M127 (Nasdala et al., 2008; Mattinson, 2010) zircon reference materials were analysed, and the results were consistently in excellent agreement with the published ID-TIMS ages. Full analytical details and the results for all quality control materials analysed are reported in Table 1 in the electronic supplementary material. The calculation of ages and plotting of concordia diagrams were performed using Isoplot/Ex 3.0 (Ludwig, 2003). Probability Density Diagrams were created using the Age Display spreadsheet of Sircombe (2004) using a 90–110% concordance filter. For all analyses reported in this study the conventional concordance was used (e.g., concordance = 100*(206Pb-238U age/207Pb-206Pb age) and analyses with a concordance between 90 and 110% have been treated as concordant. For Probability Density Plots, a discrimination threshold of 800 Ma was used. For example, if zircon analyses with an apparent 207Pb-206Pb age of >800 Ma, the 207Pb-206Pb age was used, and for zircon analyses with an apparent 207Pb-206Pb age of <800 Ma, the 206Pb-238U age was used. See Appendices 1 and 2 for more detailed information on the procedure and results retrieved.

2.2. Petrographic investigation

Eleven sandstone samples were collected for petrographic description from each lithostratigraphic unit under investigation. These samples were taken from the bases of arenaceous units within the Lower and Upper Balfour Formation, Upper Teekloof Formation, and the Lower Triassic Katberg Formation. Petrographic descriptions of sandstone samples followed the methodology of Dickinson (1985) and Pettijohn et al. (1987) noting the following features of sandstone grains: grain size, texture, roundness, sorting,

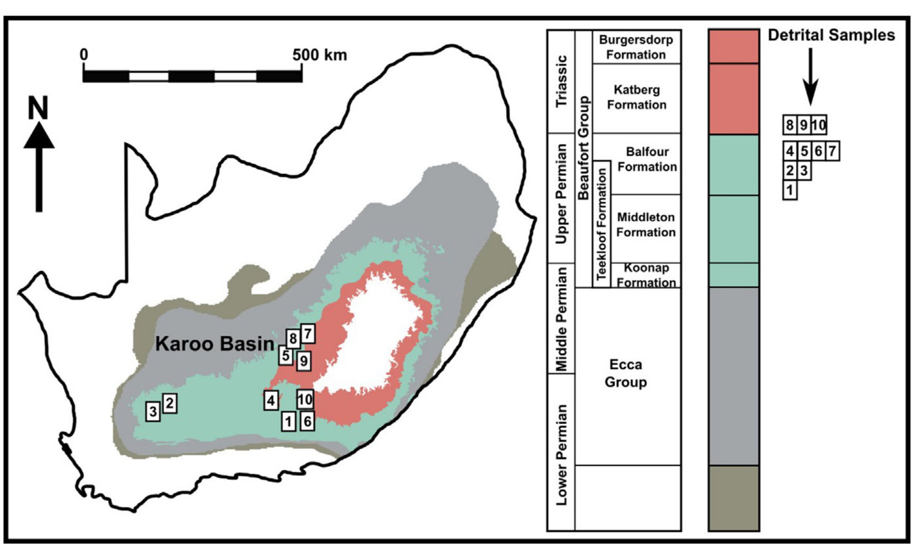

Fig. 1. A) Map of South Africa showing the localities of samples 1–10 collected in the main Karoo Basin and local geology. Proposed lithostratigraphic subdivisions of strata are correlated to the *Daptocephalus* (Balfour and Teekloof formations) and *Lystrosaurus* (Katberg Formation) assemblage zones. The stratigraphic positions of the ten sandstones sampled for detrital zircon analysis are also shown.

and maturity. This data and other interesting features such as potential ash fall particles were used to classify the sandstone petrography and interpret the provenance. Thin section descriptions were made based on quantitative observations (e.g. 250 points per thin section for mineral percentages and 50 point counts for grain size determination). Galehouse (1971) states that 250 point counts are satisfactory to obtain reliable percentages of the mineral components present. The applied point counting method used in this study was that of Dickinson (1985) and the classification scheme of Pettijohn et al. (1987) was employed (Fig. 2).

3. Results

A total of 836 concordant zircon grains were analysed from the

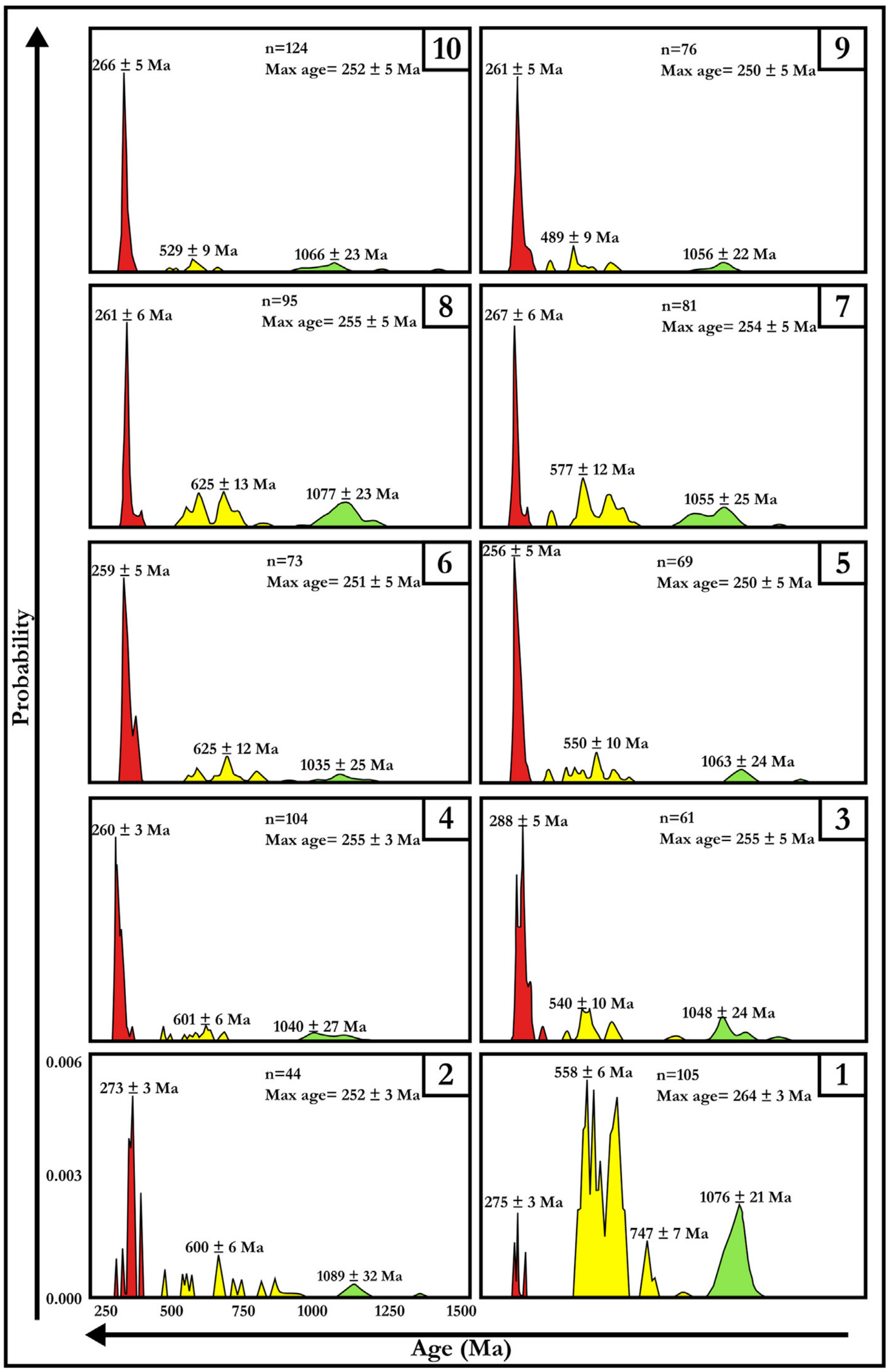

Fig. 2. Probability density plots for the 10 samples in this study. Red = the youngest zircon population (250 ± 5 Ma − 339 ± 5 Ma), yellow and green = intermediate zircon age population (364 ± 7 Ma to 878 ± 24 Ma), and (908 ± 24 Ma to 1308 ± 23). The oldest zircon populations that represent mostly Mesoproterozoic (1402 ± 26 Ma to 2450 ± 17 Ma) and Palaeoproterozoic and Archean (2756 ± 18 to 2803 ± 17) are not shown in the plots because they are trace but abundances can be seen for these populations in Table 1. A cathode luminescence SEM image of the youngest detrital zircon grain is also shown for each sample. Other grains can be viewed in supplementary material. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

10 sandstone samples (Table 1, supplementary material). The supplementary material includes all the data (including discordant grain data), Wetherill Concordia diagrams, and the cathode luminescence SEM images of all grains collected from the sandstone samples. For ease of comparison and explanation, the probability density plots from each sample are shown in Fig. 3 with SEM image of the youngest concordant zircon grain.

3.1. Detrital zircon age populations of rock samples from the Upper Permian-Lower Triassic Beaufort Group

The detrital zircon populations sampled from the Upper Permian-Lower Triassic Beaufort Group are very similar and comprise three main populations: 1) a latest Carboniferous to latest Permian-Triassic population (250 ± 5 Ma – 339 ± 5 Ma) (red section of probability denisty plot); 2) a dominantly Cambrian to Neoproterozoic population (489 ± 5 Ma to 878 ± 24 Ma) (yellow section of probablility density plot), and 3) dominantly Mesoproterozoic population (908 ± 24 Ma to 1308 ± 23) (green section of probability density plot on Fig. 2). The oldest grains in the samples are split into two groups that range from dominantly Palaeoproterozoic (1402 ± 26 Ma to 2450 ± 17 Ma) to Archean (2756 ± 18 to 2803 ± 17). These are shown in Table 1 and the supplementary material, but not in Fig. 2 because they are trace and did not show significant peaks in the probablity density plots. Most of the samples share a dominant latest Carboniferous to latest Permian-earliest Triassic zircon population (250 ± 5 Ma – 339 ± 5 Ma), ranging from 39.2% to 64% of the population (Table 1, Fig. 2). A single exception is Sample 1 from the Lower Balfour Formation which had the lowest percentage of the young population, only 4% of the total sample.

3.2. Petrography and inferred provenance

The sandstones are similar in their petrographic classification, ranging from litharenite to sublitharenite, apart from four samples which plot in the subarkose (One Lower and two Upper Balfour Formation samples) and litharenite (one Upper Balfour Formation sample) fields (Fig. 3). The sandstones of the Upper Permian – Lower Triassic Beaufort Group were plotted on a provenance ternary diagram (Dickinson, 1985) (Fig. 3), which indicates that most the samples plotted were mainly sourced from recycled orogen. However, one sample from the Lower Balfour Formation did show continental block provenance and two from the Upper Balfour Formation plotted as a mixture between continental block and recycled orogen.

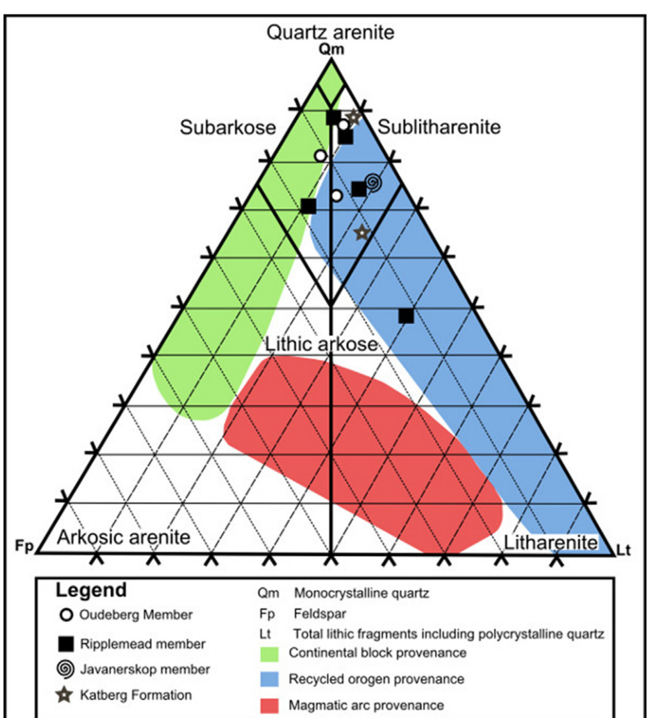

Fig. 3. Ternary diagram after Dickinson (1985) for provenance and rock classification of the 11 samples under investigation.

4. Discussion

In this section the most likely source areas for detrital zircon populations obtained from the samples collected from sandstones in the lower and upper Balfour Formation, upper Teekloof Formation, and the Katberg Formation are discussed. At the time of deposition of the Beaufort Group, southern Africa was part of Pangea (Stampfli et al., 2013) implying that source areas once available to the Karoo Basin are likely now on other continents that were once part of the supercontinent (Fig. 4). The location of the dominant source areas at various positions in the stratigraphic record of the Upper Permian and Lower Triassic Beaufort Group is useful for gaining insight into basin development of the Lopingian Karoo Basin. Secondly the maximum depositional age is used to roughly estimate the timing of deposition for the different stratigraphic units. Finally, the issues with sedimentary recycling in obscuring true provenance signatures is also discussed.

4.1. Provenance of the Upper Permian – Lower Triassic Beaufort Group

Similarities in zircon population densities between the Upper Permian and Lower Triassic Beaufort Group implies that source areas did not change significantly across the Permo-Triassic Boundary. All the upper Balfour Formation (samples 4–7) and lower Katberg samples (samples 8–10) have the same dominant population, which is the youngest population (250 ± 5 Ma – 339 ± 5 Ma). This population has Th/U ratios higher than 0.07 (supplementary material) which means they are of magmatic and not metamorphic origin (Rubatto, 2002), and are therefore likely representative of juvenile magmatic material. Results obtained from other recent workers (Vorster, 2013; Bowden, 2013) show that this young population is absent from older rocks of the Cape and Karoo basins (e.g. Cape Supergroup, Dwyka and Ecca groups). This is an interesting observation because one sample from this study (Sample 1) contains only four grains from this population. This sample is the lowest stratigraphic sample in this investigation, representing the Oudeberg Member (lower Balfour Formation). Samples 6 and 10 come from the same portion of the basin, but come from higher up in the stratigraphy and explains their major difference in population density plot. These results may mean that there was an influx of material from a new source which was introduced only in the latest Permian. Palaeocurrent investigations have identified a dominantly southerly source area for the Karoo Basin (Cole, 1992; Wickens, 1996; Cole and Wipplinger, 2001) and others predict the presence of a southern magmatic arc related to subduction of the Palaeo-Pacific Plate and formation of the Gondwanides (Johnson, 1991; Milani and De Wit, 2008).

A very small percentage of the total population in all samples have Devonian, Silurian, and Ordovician grains. Vorster (2013) and Bowden (2013) both suggest South America's Deseado Massif (DM), North Patagonian Massif (NPM), Famennian Arc, and Antarctica's Ross Orogeny as potential source areas for these grains because

Table 1
Numbers of grains collected for each sample are shown from each major geological period. Data obtained from detrital zircon dating and grain analysis are summarized in the probability density plots in Fig. 2.

Sample #	Trias	Perm	Carb	Devo	Silur	Ordo	Cam	Neo-P	Meso-P	Paleo-P	Arc
Upper Permian											
1	0	3	1	0	0	0	21	56	24	0	0
2	2	17	4	0	0	2	2	9	5	3	0
3	0	24	11	1	0	1	5	7	9	2	1
4	0	61	5	0	2	1	2	13	16	2	2
5	0	39	2	2	0	4	3	9	7	3	0
6	0	35	7	0	0	0	4	13	8	4	2
7	0	31	3	2	0	0	11	26	11	1	0
Lower Triassic											
8	0	33	4	0	0	2	8	26	20	1	1
9	1	46	8	2	0	0	7	5	6	0	1
10	0	77	7	0	0	2	5	10	18	1	4
Grain shape	Euhedral	Euhedral	Euhedral	Some crystal faces preserved	Some crystal faces preserved	Some crystal faces preserved	Some crystal faces preserved	Rounded or broken	Rounded or broken	Rounded or broken	Rounded or broken

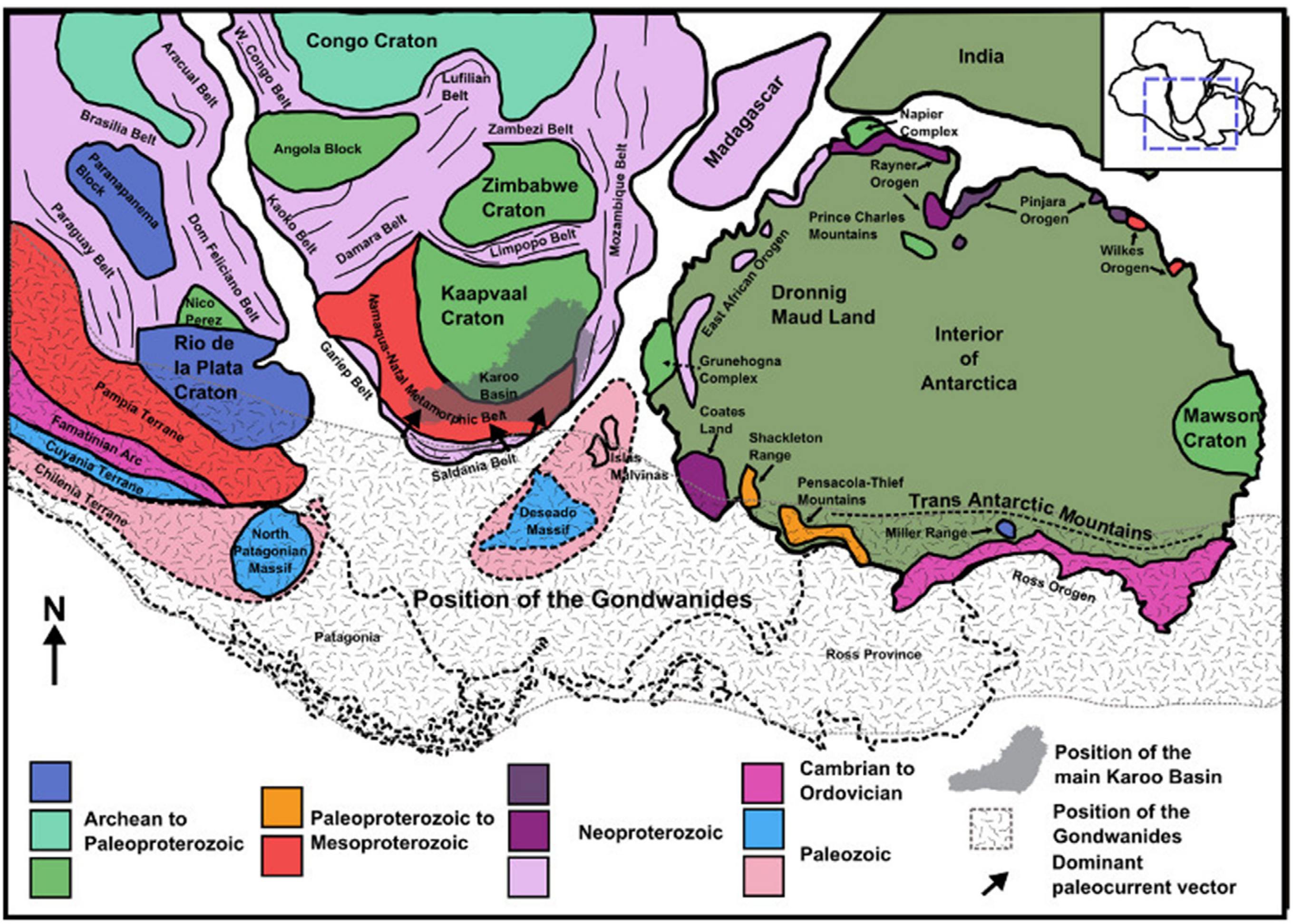

Fig. 4. Proposed position of Gondwana and the Gondwanides during the deposition of the Karoo Supergroup showing possible source areas for detrital zircons indicated by colour coded age groups. These source areas include Archean to Palaeoproterozoic cratons, Neoproterozoic mobile belts, Palaeozoic volcanics, and intrusions. Not shown is the theorized Lopingian arc volcanism south of the Gondwanides that was likely a source of dominant young detrital population sampled in this study. Modified after Pankhurst et al. (2006), Veevers and Saeed, (2013), Vorster (2013), and Bowden (2013). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

they would have been close to the southerly portion of the Karoo Basin during the Lopingian (Cole, 1992; Le Roux, 1995; Ben-Avraham et al., 1997; Pankhurst et al., 2003, 2006; Federico et al., 2006; Willner et al., 2008; Fourie et al., 2011; Tibaldi et al., 2013).

The large Cambrian-Neoproterozoic population was likely sourced from rocks associated with the Pan African Orogeny, preserved as a number of mobile or metamorphic belts, accreted terranes, and minor cratons during the formation of Gondwana (Gresse et al., 2006).

Mesoproterozoic and Paleoproterozoic zircon grains are uncommon in the samples obtained in this study and similar abundances were obtained from Cape Supergroup samples (Vorster, 2013; Bowden, 2013). The major Mesoproterozoic-early Paleoproterozoic source near the Karoo Basin is the Namaqua-Natal Metamorphic Province (NNMP), although South America's Pampia terrane may have also been a potential source (Fig. 4). The NNMP is located to the west and south of the Kaapvaal Craton and contains igneous and metamorphic rocks that are divided into several accreted terranes and subprovinces (Cornell et al., 2006; Eglington, 2006; Bailie et al., 2011). All of these terranes and subprovinces are potential source of zircons to the Cape Supergroup, which later could have also been source of sediment for the Beaufort Group.

The origin of the Archean grains is more cryptic and have also been traced in the Cape Supergroup samples of Vorster (2013) and Bowden (2013). They are probably sourced from the Transvaal Supergroup, or by reworking of older successions in the NNMP (Vorster, 2013). All the oldest grains in the samples are broken and fragmentary, pointing to a complex depositional history that very likely predates the formation of Pangea and probably were derived from other older sedimentary units, obscuring the true source areas of the Beaufort Group (Andersen, 2005; Andersen et al., 2015). Nevertheless, the Gondwanides appear to have been a significant source area for all of the rocks of the Beaufort Group, even in the distal parts of the basin where palaeocurrents are from the north-northeast.

4.2. *Sedimentary sink and LA-SF-ICP-MS*

Work conducted by Andersen (2005), Andersen (2013), Andersen et al. (2015), and Orejana et al., 2015 concluded that the current detrital zircon analytical methods assume an uninterrupted and direct pathway between a clastic sediment at its site of deposition, and its original source in crystalline bedrock. They suggest this “source to sink” relationship has been obscured in many cases by repeated events of sediment recycling. This has been alluded to by Vorster (2013) who suggested that similarities in population signatures between the Ecca Group and Cape Supergroup by a large Neoproterozoic population over a trace Archean population shows significant sediment recycling of the Cape Supergroup rocks into the Karoo Basin. This sediment recycling obscures the signal from any freshly eroded primary crystalline (Archean) basement sources (Moecher and Samson, 2006; Andersen et al., 2015). The second largest population in the 10 samples analysed is Neoproterozoic-Mesoproterozoic. Archean grains are also scarce. This implies significant sediment recycling also likely obscured primary source rocks of the Beaufort Group.

4.2.1. *Maximum age of deposition and problems with zircon dating*

The euhedral nature of the youngest zircon population which are of Permian age, and lack of Permian igneous rocks in the Karoo Basin indicates that the zircon grain origins are directly from a magmatic source and have not been significantly reworked. Evidence for this arc system, which is no longer preserved, is the presence of ashes within the Ecca and Beaufort Group (Cole, 1992; Fildani et al., 2007, 2009; Milani and De Wit, 2008; Mckay et al., 2011; McKay et al., 2015). These ashes have been dated and show a range of ages which conflict with sedimentological and palaeontological evidence on the age of the upper Beaufort Group (Fildani et al., 2007, 2009; McKay et al., 2015).

It has been suggested that this young zircon population was incorporated into upper Permian rocks by fluvial processes or by air fall during explosive volcanic activity along the arc system (Rubidge et al., 2013; McKay et al., 2015). Conflicting maximum depositional ages for the upper Beaufort Group using detrital and syndepositional zircons must mean that not all the young grains are reliable to discern the maximum age of deposition of their host rock. In this study two Triassic-aged grains were obtained from Sample 2 taken from the upper Teekloof Formation (226 ± 3 Ma; 246 ± 3 Ma), which is currently regarded as Lopingian (late Permian) on both palaeontological and radiometric dating evidence (Rubidge et al., 2013; McKay et al., 2015). Although these grains are concordant they are not regarded as a true reflection of the maximum depositional age due to several factors. These factors include Pb-loss not revealed by resolvable discordance on the concordia curve (Vorster, 2013). When one reaches the section of the concordia curve that is asymptotic, the discordia line will nearly coincide with the concordia (see Vorster, 2013 and supplementary material). In this situation, the error ellipses of individual data points will encircle both the concordia curve and the discordia curve (Vorster, 2013). Thus, Pb-loss in some grains would not be revealed by resolvable discordance within the error limits of the dating schemes used.

Additionally, the metamict nature of the detrital zircon grain also needs to be considered. The metamictization of a natural zircon results from accumulated radiation damage to the crystal structure due to radioactive decay of U and Th that substitute for Zr in the crystal structure (Holland and Gottfried, 1955; Headley et al., 1982; Woodhead et al., 1991). Additionally, metamictization can be exacerbated by extremely high U or Th content of a zircon crystal (Soman et al., 2010; White and Ireland, 2012; Deng et al., 2013). Metamict detrital zircons under cathode luminescence images include misorientated crystallites, mixed crystalline and amorphous domains, or the general loss of crystal structure (Woodhead et al., 1991; Corfu et al., 2003). The grains in question from Sample 2 do not have unusually high U or Th content however, little crystal structure is preserved. Moderate zoning is present in two of the Triassic grains (A-59, A-370) but they are not euhedral in appearance with well-developed sector or oscillatory zoning which is unusual for the young population observed in this study. Additionally, the quality of zircons retrieved from Sample 2 is exceptionally poor and thus only 44 showed concordance. Therefore, these Triassic grains are not regarded as the true maximum depositional age for Sample 2.

While previous studies (Fildani et al., 2007, 2009; Bowden, 2013; McKay et al., 2015) do not take account for any of these factors when estimating the maximum ages of deposition for their samples, this does not imply that all the young grains cannot be trusted to present an accurate maximum depositional age. The prevalence of this population in this study's samples makes it very unlikely that the young age for all of them are the result of Pb-loss. These young ages are also in agreement with the estimated Upper Permian stratigraphic age of the rocks obtained from palaeontological and sedimentological data (Rubidge et al., 1995; Smith and Botha-Brink, 2014).

5. Conclusions

This study investigated zircons from the Upper Permian Balfour, upper Teekloof, and Lower Triassic Katberg formations for possible age determination, provenance and basin development studies. Ten samples, analysed using Laser Ablation – Magnetic Sectorfield - Inductivelty Coupled Plasma - Mass Spectrometry (LA-SF-ICP-MS), revealed a dominant Late Carboniferous-Late Permian population (250 ± 5 Ma – 339 ± 5 Ma), a secondary Cambrian-Neoproterozoic (489 ± 5 Ma to 878 ± 24 Ma), a minor Mesopropertozoic (908 ± 24 Ma to 1308 ± 23), and trace Devonian, Ordovician, Proterozoic and Archean grains.

Older populations are most probably related to sedimentary recycling in the Gondwanide Orogeny and therefore are not representative of true "source to sink" or useful for provenance studies in the main Karoo Basin. However, the youngest Late Carboniferous- Late Permian population contains elongate euhedral grains, possibly representing direct ash fall input from the source since no igneous rocks of Late Permian age are present in the Karoo Basin.

Some previous detrital zircon age determination studies contradict sedimentological and palaeontological evidence on the maximum depositional age of the upper Beaufort and Ecca groups, thus proposing these rocks represent the Triassic period. These studies fail to account for factors such as Pb-loss not revealed by resolvable discordance on the concordia diagram, and metamictization of a natural zircons. However, the large amount of young grains obtained in this study implies not all are unreliable for estimating maximum depositional age of the upper Beaufort Group, and in conjunction with sedimentological and palaeontological evidence confirms an upper Permian age for the Balfour and upper Teekloof formations.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at https://doi.org/10.1016/j.jafrearsci.2017.11.006.

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