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Enhancing tectonic and provenance information from detrital zircon studies: assessing terrane-scale sampling and grain-scale characterization

JACK HIETPAS1, SCOTT SAMSON1*, DAVID MOECHER2 & SUVKANAR CHAKRABORTY2
1Department of Earth Sciences, Syracuse University, Syracuse, NY 13244, USA
2Department of Earth and Environmental Sciences, University of Kentucky, Lexington, KY 40506, USA
*Corresponding author (e-mail: sdsamson@syr.edu)

Abstract: Determining detrital zircon U–Pb ages has become the method of choice for single-mineral-based provenance studies focused on the identification of potential source regions of siliciclastic sediments. Advances in microanalytical methods have significantly accelerated the acquisition rate of U–Pb ages, thus allowing for more statistically significant zircon age datasets to be acquired than previously. However, several studies have demonstrated limitations of relying solely on detrital zircon as a provenance proxy. To further assess the utility of this provenance indicator we measured U–Pb ages of detrital zircon derived from modern sediment collected from the French Broad River and its tributaries that drain portions of the Appalachian Orogen in southeastern USA. The results demonstrate that significant detrital zircon age variations occur along the length of the river. The age variations suggest that characterization of entire sedimentary formations by analysis of single samples may be misleading and that a multiple-sample approach is required. In addition, by incorporating high-magnification cathodoluminescence images with Th/U for each detrital grain, a more robust interpretation can be made regarding zircon source.

Supplementary material: Global positioning system coordinates for each sampling location and complete zircon data are available at http://www.geolsoc.org.uk/SUP18445.

Sedimentary provenance analysis has the potential to provide constraints on source lithology, palaeogeographical reconstructions (Murphy et al. 1996), sedimentary basin evolution (Schmitt & Steidtmann 1990), stratigraphic correlation (Stattegger & Morton 1990), timing of sediment deposition (Dickinson & Gehrels 2009; Rainbird et al. 2001), tectonic setting (Dickinson & Suczek 1979; Dickinson et al. 1983; Dickinson 1985), rates of regional uplift (Bernet & Spiegel 2004), and delineation of ancient sediment dispersal systems (Rainbird et al. 1992). Accurate identification of source lithology is limited by the specific physical and chemical properties of each detrital mineral during weathering, transportation, lithification, response to diageneisis, and metamorphism (Morton & Hallsworth 1999). In addition, the sediment transport–depositional system (fluvial, aeolian, littoral, etc.) and climate strongly influence sediment composition (Suttner et al. 1981). Inferring sediment provenance is further obscured as the detrital mineral spectrum continually evolves through transport, deposition, and diageneisis (Weltje & von Eynatten 2004). Given all these potential pitfalls in determining sediment provenance, it would be prudent to exploit all available provenance information recorded in the sedimentary record for such investigations to be successful.

In spite of the numerous approaches to, and assumptions inherent in provenance studies, the discipline has moved rapidly toward single-mineral and single-sample-based approaches. Such studies have, for example, utilized density (i.e. heavy mineral types), colour and morphology (Lihou & Mange-Rajetzky 1996), crystallization ages (Gehrels et al. 2008), and mineral chemistry (e.g. Zack et al. 2004; Morton & Yaxley 2007). Utilizing characteristics of specific minerals as a provenance tool, as opposed to bulk rock or bulk alluvium, minimizes the effect of the differing diagenetic stabilities and hydraulic sorting properties of minerals (Morton 1985; Mange & Maurer 1992). The trend toward examining single detrital mineral phases rather than bulk-rock approaches has been considered the single most important development in determining sediment provenance (Morton 1991).

Detrital zircon geochronology has received particular attention as a single-mineral provenance tool because the abundance of zircon (in terms of numbers of grains) does not change drastically during sediment transport, owing to the inherent stability of zircon. The proliferation of zircon-based studies has largely been driven by advances in high-throughput in situ microbeam methods, such as laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) and secondary ion mass spectrometry (SIMS). The time required to determine the crystallization age of zircon has changed from the scale of hours using thermal ionization mass spectrometric techniques to minutes using LA-ICP-MS. With access to such rapid techniques, sediment provenance investigations have been performed on a scale previously unattainable. However, with rapid data acquisition there is a tendency to omit characterization of each detrital crystal prior to analysis. This is most evident in detrital zircon studies via LA-ICP-MS that neglect high-magnification cathodoluminescence (CL) imaging of each grain prior to age measurement. Although groups of grains in a sample mount may be imaged via CL for zoning, characteristics of single zircon crystals are typically not considered in provenance studies and ‘cores’ are typically analysed. CL imaging, essentially zircon petrography, provides valuable information concerning zircon growth histories for high-resolution geochronology of magmatic and metamorphic events (Hanchar & Miller 1993; Hanchar & Rudnick 1995; Connelly 2000; Hoskin & Black 2000; Corfu et al. 2003). In the latter type of studies, the utility of CL imaging is widely accepted as a requirement for interpretation of ages. Crystals with complex histories reflecting multiple stages of
Detrital zircon has proven to be a powerful provenance tool in understanding ancient aeolian processes (Soreghan et al. 2002), palaeo-drainage patterns (Rainbird et al. 1992), terrane discrimination (Samson et al. 2005), and palaeogeographical reconstructions (Gaudette et al. 1981; Ross & Bowring 1990; Cawood & Nemchin 2001). Despite these successes, zircon-based provenance studies have limitations. Failures of zircon-based provenance studies to fully identify all source terranes for sediments under investigation are particularly evident in studies of Palaeozoic clastic sequences in the Appalachian orogen. Detrital zircon age distributions in all such sequences are dominated by recycled Grenvillian plutons. One might not recognize formation of the supercontinent Pangaea (e.g. Hatcher 1989). Numerous detrital zircon provenance studies of Appalachian foreland clastic sequences have attempted to discern the kinematics of Neoproterozoic post-Rodinian rifting, early Palaeozoic passive margin development, and Palaeozoic assembly of the orogen (Bream et al. 2004; Thomas et al. 2004a; Becker et al. 2005; Cawood et al. 2007; Park et al. 2010).

The studies of Gray & Zeitler (1997) and McLennan et al. (2001) were the first to reveal the inability of detrital zircon to fully record the defining tectonic events of the Appalachian Orogen. Detrital zircon age populations from the Silurian Shawangunk Formation consist entirely of Grenville ages (c. 1300–950 Ma) with no Taconic (c. 470–440 Ma) signal, a surprising result given its Silurian depositional age and late synorogenic character.

The zircon age spectrum from the Upper Carboniferous Pottsfield Formation records signals for both the Grenville and Taconic events but exhibits a very limited Acadian zircon signal, despite the formation having a depositional age that is at least 55 Ma younger than the main phase of Acadian orogenesis (Gray & Zeitler 1997). In contrast, detrital micas from this formation record Acadian age spectra, demonstrating that Acadian source terranes were exposed during deposition of the Pottsfield Formation.

The detrital zircon spectrum from the Middle Ordovician Austin Glen Member of the Normanskill Formation records a strong Grenville signal but lacks a 470–440 Ma zircon signal, signifying that the syntectonic Taconic orogenic event was not captured. The detrital zircon age spectrum of Devonian red beds from the Catskill clastic wedge, a syn- to post-Acadian unit, exhibits a bimodal zircon age distribution with strong signals recording Grenville and Taconic events, but no Acadian ages (c. 420–380 Ma). Here again, detrital zircon age spectra fail to record syntectonic events and are dominated by recycled Grenville zircon (McLennan et al. 2001).

Detrital zircon ages were determined in eight Upper Carboniferous sandstones and conglomerates collected in the foreland basin along c. 600 km of strike of the Alleghanian orogenic front (Thomas et al. 2004a; Becker et al. 2005). Detrital zircon in these clastic rocks, deliberately sampled to include a wide range of lithologies, are dominated by Grenville-age zircon populations, have limited Taconic and Acadian signals, and completely miss the signal from the presumed Himalayan-scale continental collision during the Alleghanian Orogeny.

Detrital zircon ages were also measured from six modern Appalachian rivers (Eriksson et al. 2003). The detrital zircon age spectra are again dominated by Grenville-aged zircon with minor Taconic and Acadian signals, despite Grenville basin accounting for only 12% of the exposure in this part of the southern Appalachian Orogen. Detrital zircon completely failed to record Alleghanian ages in 67% of the rivers sampled. In total, five Alleghanian-aged zircon crystals were recorded from the pooled c. 600 age determinations, yielding a 0.8% success rate for zircon to capture the c. 300 Ma event. This is particularly surprising as two of the sampled rivers (Savannah and James) are directly draining source regions known to have exposed Alleghanian-aged plutons.

**Petrogenetic factors**

If interpretation of the tectonic history of the southern Appalachian region was based on detrital zircon age populations as the sole proxy for identifying all potential sources for Palaeozoic clastic sediment or modern alluvium, one might conclude that there had been only two significant prior collisional events and one might not recognize formation of the supercontinent Pan-
gaea. Clearly, the incomplete tectonic record of Appalachian detrital zircon would be considered a major limitation for producing accurate tectonic models for eastern Laurentia.

Several factors explain the shortcomings of provenance studies based solely on detrital zircon age distributions (Moecher & Samson 2006). First, new zircon (cores or rims) is not produced in response to all magmatic or tectonic events. Emplacement of zircon-bearing magmatic rocks is most commonly observed in orthogonal convergent plate settings. However, if a major tectonic event is dominated by strike-slip or transpressional plate motions (e.g. Acadian deformation effects in the southern Appalachians: Adams et al. 1995; Merschat et al. 2005), the event may be primarily defined by regional metamorphism rather than by magmatism. Zircon might record this thermotectonic event by either substantial Pb loss (Kirkland et al. 2007) or by the formation of new zircon crystals. Zircon growth generally requires upper amphibolite to near-anatectic conditions (Hoskin & Black 2000). If a tectonic event was not associated with high-grade metamorphism, few zircon crystals would be formed, rendering this event invisible to the detrital zircon record.

The second potential problem is zircon inheritance in newly generated magmas. Zircon has an extremely low solubility in metaluminous and peraluminous melts (Watson & Harrison 1983). Consequently, a magma generated at relatively low temperatures can incorporate a significant amount of xenocrystic zircon that can serve as nucleation ‘seeds’ for new zircon growth of younger age (Miller et al. 2003; Chappell et al. 2004). Zircon shed from igneous bodies that contain significant inheritance will appear to be derived from an older source when cores are exclusively analysed. That older source may then be erroneously located in palaeogeographical and tectonic reconstructions. However, analysis of magmatic overgrowths would demonstrate that the zircon was shed from a terrane characterized by a younger magmatic event.

A third potential problem is that zircon is an ultra-stable mineral in terms of its resistance to chemical and mechanical weathering (Mange & Maurer 1992). As a consequence, zircon can undergo repeated cycles of erosion, transport and lithification into stabilized sedimentary and (subsequently) metasedimentary terranes. The effect of zircon recycling is a natural bias in the detrital record, resulting in an over-representation of older ages rather than those that best reflect the majority of detritus that is being shed into the basin under investigation.

Geological setting

The French Broad River and its tributaries in North Carolina and Tennessee were chosen because they drain the tectonically complex and lithologically diverse southern Appalachian Orogen (Fig. 1). The French Broad River is undammed along the length sampled, so sediment transport has been largely undisturbed since Cenozoic formation of the drainage basin. The primary potential zircon donor sources drained by the French Broad River and its tributaries include (1) Grenville basement, consisting of ortho- and para-gneisses crystallized and metamorphosed at c. 1300–900 Ma; (2) the Ashe–Tallulah Falls metamorphic suite, a mixed assemblage of late Neoproterozoic metapelites and mafic volcanic rocks metamorphosed to upper amphibolite facies during Taconian orogenesis (Merschat & Weiner 1988); (3) the Henderson Gneiss, an Ordovician metagranite deformed during middle Palaeozoic orogenesis; (4) late Neoproterozoic continental rift volcanic and plutonic rocks. All these components were, to varying degrees, affected by subsequent Acadian and Alleghany orogenic events.

Methods

Fourteen alluvium samples were collected from the French Broad River from its headwaters to the point at which the river enters an artificial impoundment (Fig. 1). An additional sample was collected from the Pigeon River, the main tributary of the French Broad river in the study area. Approximately 5 kg of alluvium
was collected at each location. With the exception of coarse sieving (c. 5 mm) no attempt to concentrate the samples was made at the sampling locations. In addition to the alluvium samples, the North Carolina Geological Survey (NCGS) provided alluvium collected from six sites along minor tributary streams within a 150 km² area of Mesoproterozoic (Grenville) and Neoproterozoic (Ashe–Tallulah Falls metamorphic suite) basement rocks. Some of these tributaries flow into the Pigeon River, which then empties into the French Broad River (Fig. 1). Three samples (CT 136, 145, 147) were collected in small drainages that accumulate alluvium from only Ashe–Tallulah Falls metamorphic suite bedrock, and three samples were collected from larger drainages that accumulate alluvium from Ashe–Tallulah Falls metamorphic suite and Grenville basement lithologies. The tributary samples were coarse-sieved and panned at the collection site to concentrate heavy minerals. Dense minerals were then separated from the concentrates using bromoform ($\rho = 2.85$ g cm$^{-3}$) (Merschat & Weirich 1988).

Alluvial samples from the main trunk of the French Broad River were sieved using disposable polyester sieve cloth with 500, 250, 100 and 50 µm openings. Wilfley table concentrates for the 250–100 µm and 100–50 µm fractions were further purified using acetylene tetrabromide (TBE, $\rho = 2.95$ g cm$^{-3}$). The <50 µm size fraction was placed directly into 50 ml polyethylene screw-capped tubes containing TBE and centrifuged at 700 r.p.m. for 10 min. This method of dense mineral purification was used to minimize the loss of the fine-grained component often encountered with a Wilfley table. Dense minerals were then magnetically separated using a Frantz model LB-1 isodynamic separator at 10° forward and 20° side slopes. Zircon crystals were selected from the 1.5 A non-magnetic fraction.

A total of 100–150 zircon crystals were selected for analysis using stereomicroscopy at magnifications of 10–80X. Crystals were chosen to represent all recognizable populations based on size, crystal morphology, colour and inclusions. The focus of our selection was to capture the maximum number of potential zircon donor regions, not to attempt to obtain a random zircon population.

Two detrital zircon sampling strategies are used in most studies: (1) a method striving to obtain a ‘random’ sampling of the zircon separate, or (2) a directed approach that attempts to identify all potential age populations. Both methods have limitations. The random sampling strategy, which involves shaking zircons from a vial of concentrate onto adhesive tape, may be advantageous for correlating sedimentary units because the influence of analyst-induced selection bias is minimized. However, random sampling produces a natural bias in the age spectra of detrital zircon towards crustal regions that were the most prolific in zircon production (i.e. most zircon fertile), which might not reflect the areal importance of those sources (Moecher & Samson 2006; Dickinson 2008). If the goal of a provenance study is to maximize the recognition of potential source regions then the study should incorporate a directed sampling strategy toward all zircon populations (e.g. Gehrels & Dickinson 1995; Fedo et al. 2003; Andersen 2005), thus increasing the likelihood of recording a maximum number of sediment donor regions rather just the most zircon-fertile regions.

Selected single zircon crystals and age standards were aligned in a grid pattern on 3M™ packaging tape and embedded in Buehler Epothin™ low-viscosity epoxy. After 24 h of curing at c. 40°C, mounts were ground to expose the interiors of the crystals using 2000 grit sandpaper. A final polish using 1 µm aluminium oxide was then performed. Each zircon crystal was imaged by high-magnification backscatter-electron (BSE) and CL to reveal internal details including potential cores and rims (Corfu et al. 2003, and references therein).

The majority of the U–Pb dating of detrital zircon was performed by LA-ICP-MS using the Isotope-Probe® at the Laser-Cron Center at the University of Arizona (Gehrels et al. 2008). Common Pb corrections were made by measuring 204Pb and assuming common Pb isotopic composition as modelled by Stacey & Kramers (1975). The U/Pb and 206Pb/207Pb fractionation is calibrated relative to fragments of a Sri Lanka zircon standard (SL-1) with an age of 564 ± 3.2 Ma (2σ) (Gehrels et al. 2008). This standard is also used to calibrate the U concentration and Th/U of unknowns, and is accurate to 20%. Decay constants ($\lambda_{235U} = 8.9485 \times 10^{-10}$, $\lambda_{238U} = 1.5525 \times 10^{-10}$) and isotopic abundance ($^{238}U/^{235}U = 137.88$) follow those proposed by (Steiger & Jäger 1977). Analyses that are >30% normally discordant (as determined by 206Pb/238U and 206Pb/207Pb* dates) or >5% reversely discordant are not reported. The reported dates for crystals younger than 900 Ma are 206Pb*/238U dates and for crystals older than 900 Ma are 206Pb*/207Pb* dates. The laser spot size was either 25 or 35 µm depending on grain size or crystal domain (core v. rim).

In addition to the LA-ICP-MS data, 46 analyses of zircon from the tributaries were performed using the CAMECA IMS 1270 ion microprobe at the University of California, Los Angeles (UCLA) following methods described by Grove et al. (2003). Zircon standard AS3 with an age of 1099 ± 1.2 Ma (Paces & Miller 1993; Schmitz et al. 2003) was used for ion microprobe analyses. All reported zircon data (LA-ICP-MS and SIMS) have been given by Hietpas et al. (2010).

Results
French Broad River alluvium

Detrital zircon age distributions for 10 samples of French Broad River alluvium, one sample of Pigeon River alluvium, and the entire alluvium suites are shown in Figure 2. Sample FB2, collected in the headwaters region draining an area underlain by diverse Precambrian and Palaeozoic lithologies, exhibits primarily Mesoproterozoic zircon ages (1300–900 Ma), with a few Neoproterozoic and Palaeozoic ages. All but two of the 59 Mesoproterozoic grains have more variable Th/U of 0.30–1.57. The Palaeozoic and Neoproterozoic grains have Th/U ranging from a high of 1.51 to essentially zero. In marked contrast, the next two samples downstream (FB3 and FB4) are dominated by zircon with Ordovician ages with only a few Mesoproterozoic ages. These two samples were collected where the French Broad River flows over the mapped extent of the Henderson Gneiss. These zircons have Th/U values that range from 0.36 to 1.48. From FB1 to FB10 the zircon with Mesoproterozoic age components begin to reappear and eventually dominate the age spectra, with a variable component of Ordovician ages, and rare Neoproterozoic and early Mesoproterozoic to Palaeoproterozoic ages. Relatively strong Ordovician peaks persist in samples FB1 and FB5, collected where the French Broad flowes across the Ashe–Tallulah Falls metamorphic suite. Sample FB8, collected in the area underlain solely by Mesoproterozoic basement, contains only a few grains with Ordovician ages. Two well-resolved Mesoproterozoic age peaks at c. 1050 and 1180 Ga are present in samples FB13 and FB15. A few Neoproterozoic ages are present in several of the samples. A compilation of all French Broad River zircon analyses (n = 1102) has a dominant Ordovician peak at 450 Ma and two broad Mesoproterozoic peaks at 1050 and 1175 Ma (Fig. 2).
The age spectrum of detrital zircon cores in alluvium collected from the tributaries draining an area underlain solely by Ashe–Tallulah Falls metamorphic suite metapelites and Mesoproterozoic orthogneiss is shown in Figure 3a. The major age peak is broadly Grenvillian with two subgroups at c. 1050 and 1175 Ma, with a minor Ordovician age peak and scattered Neoproterozoic ages. The ages of zircon rims, identified from CL images for each grain, are solely Middle Ordovician. Combining the ages of the zircon rims with those determined from cores considerably increases the size of the Ordovician age peak compared with the Mesoproterozoic peak (Fig. 3b).

Discussion

CL imaging, Th/U and geochronology

Significant additional provenance information was gained in several instances because CL imaging was performed on every grain and used as a guide for LA-ICP-MS analysis. In addition, Th/U was collected for each analysis as this ratio can be utilized as a proxy for zircon petrogenesis. Th/U values >0.5 are generally considered to be indicative of an igneous origin, whereas ratios of ≤0.1 are generally considered indicative of a metamorphic petrogenesis (Hoskin & Black 2000; Rubatto 2002; Hoskin & Schaltegger 2003). Figure 4a is a CL image for a detrital zircon crystal collected from one of the tributaries. The date of the core of this crystal is 1165 ± 56 Ma, with a Th/U
shows two growth zones. The core of the crystal yields a date of 1184 ± 20 Ma and Th/U of 0.20. The complexly zoned, but presumed magmatic rim, yielded a date of 409 ± 11 Ma and Th/U of 0.40. The identity of the source lithology is again probably not Grenville basement, and is more probably an Acadian-aged magmatic source contaminated with Grenville xenocrysts.

In Figure 4d the core of the crystal yields a Grenville date (1137 ± 45 Ma, Th/U = 0.29) but the rim date is 318 ± 8 Ma with Th/U = 0.009. This single dark, unzoned, probably metamorphic overgrowth is the only evidence obtained (from 1102 detrital zircon grains analysed) for the supercontinent-forming Alleghanian Orogeny. Without detailed CL imaging it is highly improbable that this zircon component would have been recognized.

Figure 4e and f shows CL images of zircon crystals isolated in samples FB4 and FB1, respectively. Both crystals have similar Taconic dates (453 ± 16 Ma and 466 ± 16 Ma). The CL pattern for the zircon from FB1 (Fig. 4e; dull, dark, unzoned) suggests a metamorphic origin, perhaps by dissolution and reprecipitation. This inference is supported by the very low Th/U ratio (0.006). The grain in Figure 4f, however, displays oscillatory zoning and a Th/U ratio of 1.30, features consistent with the crystal having an igneous origin. Integrating CL imaging with Th/U ratios allowed two grains with indistinguishable Ordovician dates to be identified as coming from different sources.

Nature of zircon recycling

Based on 1:24 000 scale bedrock mapping, the source region sampled by the tributaries consists of c. 40% Mesoproterozoic layered biotite gneiss and c. 60% migmatitic pelitic schists and gneisses of the Ashe–Tallulah Falls metamorphic suite. The latter comprises an extensive tract of metaclastic rocks of the Eastern Blue Ridge terrane, inferred to have been deposited in the Neoproterozoic (c. 600–700 Ma depositional age: Chakraborty et al. 2010) on oceanic crust outboard of the Laurentian rifted margin. The Eastern Blue Ridge is interpreted to have been thrust onto the Laurentian margin during early Ordovician convergence but prior to the peak of Taconian regional metamorphism. In spite of the Ashe–Tallulah Falls metamorphic suite making up the greater proportion of potential source lithologies for the tributary streams, only 7% of the detrital zircon (cores) analysed from the tributary samples are Ordovician and of metamorphic origin. Analysis of (metamorphic) zircon rims increases the proportion of Ordovician zircon ages to c. 10%, but the Mesoproterozoic ages still dominate the age spectrum. If the total of c. 200 core ages represented, for example, detrital zircon ages derived from two rock samples (assuming 100 grains per rock sample), then the inference might be made that the immediate source of sediment was dominantly Mesoproterozoic magmatic rocks and not Ordovician metamorphic rocks. The abundance of Mesoproterozoic magmatic zircons in the Ashe–Tallulah Falls metamorphic suite metapelites argues for an ultimate Grenville source of Neoproterozoic Ashe–Tallulah Falls metamorphic suite clastic protoliths, and the two extensive units together constitute a single detrital zircon source terrane in spite of tectonic models that have the Ashe–Tallulah Falls metamorphic suite being an allochthonous package of rocks (Hatcher et al. 2004). However, recycling of abundant Grenville zircon has imprinted a Laurentian identity on the Ashe–Tallulah Falls metamorphic suite, in spite of the latter being nearly 0.5 Ga younger than the Grenville.

The impact of zircon recycling on source interpretation has been noted in other modern depositional systems. Analysis of
detrital zircon in modern beach sand along the eastern Australian coast revealed a significant 700–500 Ma age component (Sircombe 1999), implying that a terrane of that age was supplying magmatic zircon to the littoral deposits. However, there is at present no terrane of that age in the immediate source region of the sediments accumulating on the Australian Pacific margin. The likely immediate source of the Neoproterozoic zircon is the Middle Triassic Hawkesbury Sandstone in the Sydney Basin. The closest original source rock is at present 7000 km away, and cannot serve as an active source of the modern beach sands.

Significant detrital zircon recycling was also demonstrated in the modern Indus and Ganges River systems (Campbell et al. 2005). The percentage of recycled zircon crystals was quantified by incorporating a He–Pb double dating approach. The results showed that recycled detrital zircon crystals represented between 60 and 70% of the zircon populations isolated from the alluvial samples (i.e. they could not have been derived from immediate sources of alluvium).

**Impact of single v. multiple samples in source identification**

The age and lithological character of bedrock currently drained by the French Broad River have an important influence on measured detrital zircon ages as a function of distance along the river’s course. Sample FB2 was collected in the headwaters region where tributaries collect sediment from an area underlain primarily by Ashe–Tallulah Falls metamorphic suite metasediments, with lesser amounts of Mesoproterozoic basement (Hatcher et al. 2004) and Palaeozoic granitoids (Miller et al. 1997, 2000). Although the location of FB2 is on the c. 450 Ma (Moecher et al. 2010) Henderson Gneiss, Mesoproterozoic ages dominate the spectrum (Fig. 2). As was demonstrated for the tributaries downstream (Fig. 3), the Mesoproterozoic peak in the spectrum for FB2 probably represents zircon recycled through the Ashe–Tallulah Falls metamorphic suite, and not direct derivation from Mesoproterozoic orthogneisses.

The French Broad River detrital zircon age spectrum changes markedly downstream of the headwaters region. Samples FB2 and FB3 are only c. 13 km apart, yet their detrital zircon age populations are significantly different (Fig. 2). FB2 has a predominance of Grenville-aged zircon, but also shows moderate peaks for both the Taconian (Middle Ordovician) and Acadian (Devonian) orogenies. However, FB3 is heavily dominated by Taconian ages, and has only a minor Grenville signal.

Sample FB3 is similar to FB4, which was also collected on Henderson Gneiss (Fig. 2). The most likely major source for FB3 and FB4 is the Ordovician Henderson Gneiss itself, which has a protolith age of 450 ± 5 Ma (Moecher et al. 2010), and zircon grains in the gneiss have Th/U of 0.45–1.14. The few Mesoproterozoic ages in FB3 and FB4 could correspond to a xenocrystic component in the Henderson orthogneiss or are simply derived from upstream Grenville sources.

The detrital zircon spectrum for sample FB1 is also heavily dominated by Middle Ordovician-aged grains. The river at this location is flowing over pelitic schists and gneisses of the Ashe–Tallulah Falls metamorphic suite. The abundance of Ordovician ages contrasts with the dominance of Grenville zircon ages observed in the alluvium of the small tributaries to the French Broad River that also drain the Ashe–Tallulah Falls metamorphic suite. Values of Th/U for FB1 grains range from 0.50 to 0.80, consistent with a Henderson Gneiss source.

Samples FB6 and FB8 are c. 13 km apart, yet the detrital zircon age spectra from these two samples are significantly different. The primary age mode for sample FB6 is Taconian, but detrital zircon ages also record minor Neoproterozoic and moderate Grenvillian signals. However, for FB8, Grenvillian ages account for 98% of the detrital zircon age population, in stark contrast to nearby sample FB6. Detrital zircon ages, at this sampling location, record no Neoproterozoic signal and only a single zircon grain records the Taconic Orogeny.

A single zircon with an age of 1.8 Ga was noted in sample FB6. This is not a typical ‘Grenville’ age, but recent Nd and Pb isotope analysis on Blue Ridge basement points toward older crustal components present among the diverse lithologies forming the crust in this area (Fisher et al. 2010) and thus there may be exposures of this age component in the immediate drainage area of sample FB6.

A modest Ordovician zircon signal reappears in samples FB10 and FB11 as metaclastic rocks of the Neoproterozoic Ocoee Supergroup appear as the local bedrock source. The Ocoee Supergroup is a thick (c. 15 km) sequence of greenschist- to upper amphibolite-facies metasiltstones, quartzites and conglomerates derived from erosion of Mesoproterozoic Grenville basement (Hadley & Goldsmith 1963; Chakraborty et al. 2010), deposited in the Neoproterozoic, and metamorphosed in the Middle Ordovician. Rocks within the Ocoee Supergroup are mainly at greenschist facies in the area of FB10 and FB11, thus it is unlikely that new zircon growth would have occurred in these local source rocks. Previous work has shown that the Ocoee is dominated by Mesoproterozoic detrital zircon (Bream et al. 2004; Chakraborty et al. 2010), with minor Palaeoproterozoic and Neoproterozoic zircon. The Ocoee is thus similar to the Ashe–Tallulah Falls metamorphic suite in many aspects, although volcanic and mafic rocks are absent in the Ocoee. Values of Th/U for the Ordovician zircons in FB10 and FB11 are 0.01–0.58, with most >0.40. The higher values are recorded in grains that were probably sourced upstream in the Henderson Gneiss. Also present are a few Neoproterozoic-aged magmatic (based on Th/U) zircon crystals. These are probably also derived (i.e. recycled) from the Ocoee, which contains a minor component of Neoproterozoic rift-related zircon grains (Chakraborty et al. 2010).

Sample FB13 was collected in an area of Cambro-Ordovician quartzite–carbonate bedrock, which includes the mature Upper Cambrian Chilhowee Group quartz arenites deposited on the Laurentian passive margin. FB13 exhibits the most diverse age components of all the French Broad River samples. In addition to the two Mesoproterozoic age populations at 1050 and 1175 Ma, and the single Ordovician population, other ages present include a few Neoproterozoic grains, a group of grains at c. 1600 Ma, and a single grain at 2000 Ma. The last is the oldest age measured in this study. The most likely immediate source of the diverse ages in alluvium sample FB13 is recycling from local Chilhowee bedrock.

Sample FB15 was collected from the Pigeon River, which is the largest tributary to the French Broad River. The Pigeon flows approximately parallel to the French Broad but it drains a greater area of Ocoee Supergroup and extends as far SE as the Ashe–Tallulah Falls metamorphic suite, where both are at sillimanite to kyanite grade and are locally migmatitic. The headwaters of the Pigeon River also collect sediment from some of the tributary streams whose zircon ages are included in Figure 2. The sample was collected just above the confluence with the French Broad River and on Cambro-Ordovician siliciclastic bedrock units. The detrital zircon spectrum for this sample is again heavily dominated by two Grenville age populations, exhibits a weak Ordovician signal, and contains two grains at c. 650 Ma. Values
et al. basement character (Cawood samples collected along the Frankland River in southwestern modern river sediments. The detrital zircon ages from alluvial in detrital zircon ages has been observed in other studies of French Broad alluvial system. Significant longitudinal variation function of immediate source area is not a unique feature of the The dramatic change in the detrital zircon age populations as a Sediment heterogeneity: examples from other modern settings

The dramatic change in the detrital zircon age populations as a function of immediate source area is not a unique feature of the French Broad alluvial system. Significant longitudinal variation in detrital zircon ages has been observed in other studies of modern river sediments. The detrital zircon ages from alluvial samples collected along the Frankland River in southwestern Australia, for example, correlated closely with the changing basement character (Cawood et al. 2003).

Detrital zircon age populations from modern river alluvium collected by Link et al. (2005) in the Snake River system, western USA, also showed significant longitudinal variation. The researchers were able to generate a record that accurately recorded all major zircon populations present in eastern Idaho and Wyoming, but only by combining data from multiple sample locations.

Lawrence et al. (2010) sampled five sub-environments from a single Amazon River sand dune. The detrital zircon age populations from these samples showed significant differences in the presence or absence of entire age populations. Based on those data, Lawrence et al. (2010) concluded that a single sand sample may not reliably characterize the sediment as a whole.

In contrast to the alluvial settings examined in the studies cited above, Sircombe (1999) measured U–Pb ages of detrital zircon crystals from 10 modern beach sands spanning c. 2000 km of the eastern coast of Australia. It would be expected that long-lived coastal high-energy depositional environments would result in efficient mixing and homogenization of clastic sediments being delivered by alluvial systems from diverse source terranes (e.g. Ingersoll 1990; Ingersoll et al. 1993; Critelli et al. 1997). However, the detrital zircon age spectra from coastal Australia also showed strong regional variation. The detrital zircon age populations were heavily influenced by local bedrock and influx by feeder tributaries, and were not well homogenized. These results further emphasize the danger of analysing detrital zircon from a single sandstone and assuming that the ages are fully representative of the entire formation.

Conclusions

The determination of sediment provenance (both modern and ancient) is a challenging task that must take into account the sedimentological properties of detrital minerals in terms of their chemical and mechanical weathering during erosion, sediment transport, lithification, and diagenesis or metamorphism. By allowing the characteristics of each grain to guide the measurement of U–Pb ages of detrital zircon crystals, a more complete and accurate assessment of the source lithologies for the sediment under investigation was obtained. Younger rim ages were frequently determined for detrital zircon crystals, demonstrating the survivability of zircon overgrowths in a moderate-energy fluvial transport system. Evidence of the Alleghanian Orogeny would have been completely missed if rims had not been identified by CL prior to analysis. The results of this study reinforce the importance of high-magnification (i.e. single-grain) CL imaging, as opposed to low-magnification imaging of tens of grains at a time, in recognizing and capturing all available age information recorded by detrital zircon.

This study has shown that alluvium collected along the length of the French Broad River is much more heterogeneous than generally thought. By investigating modern river sediments, it has been shown that the extreme heterogeneity of the detrital zircon age populations along the length of the river are due to complex and often unpredictable changes and mixing of sediment donor regions. The data have significant implications for investigators attempting to determine the provenance of ancient lithified sediment. This is particularly important when the source lithologies for a basin are not known or no longer exist. The results from this study demonstrate the need for a multiple sampling strategy that takes into account the complexities of the sediment transport system under investigation. The tectonic conclusions from studies relying on detrital zircon ages from a single sample of a sedimentary formation might be considerably biased or at least incomplete. By utilizing extremely high-throughput methods such as LA-ICP-MS, multiple samples of an ancient sedimentary formation can be characterized by zircon U–Pb ages. Combining detailed CL imaging with Th/U data on multiple sedimentary samples allows for the generation of higher resolution datasets that offer the potential for more accurately defining the nature of sediment donor regions. Such information is critical to the construction of accurate and robust tectonic models.

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