Chapter 14. Thermochronology on sand and sandstones for stratigraphic and provenance studies

Andrew Carter

Department of Earth and Planetary Sciences, Birkbeck, University of London, Malet Street, London WC1E 7HX, U.K.

a.carter@ucl.ac.uk

Clastic detritus preserved within a sedimentary basin represents a natural reservoir of geological information that can be used to constrain sediment deposition age and develop a picture of the sediment routing system and source terrain(s) in terms of location, age, composition and tectonic and climate stability. This chapter charts the development and applications of fission track analysis to solve stratigraphic and provenance problems. Many of the interpretative tools and strategies developed for fission track data are also applicable to detrital (U-Th)/He and other geochronological data. Provenance interpretations based on double and triple-dating strategies may be further improved by combining with mineral trace element data.

14.1. Historical Background

The goals of early provenance studies were much the same as today. In addition to constraining sediment depositional age, studies employed a set of samples through a stratigraphic sequence in a basin to reconstruct changes in the sediment routing systems, make stratigraphic correlations, define inputs from different source areas, and reconstruct the uplift and erosion histories of the source rocks. The first fission track papers appeared in the late 1970's whereas (U-

Th)/He provenance studies did not arrive until after 2000 (e.g. Rahl 2003), and for this reason most of the interpretative developments stem from fission track thermochronometry.

The first fission track dating papers appeared in 1964 (Fleischer et al. 1964; Fleischer et al. 1965) but the standard fission track methodology, as practiced today, only came into being following technical advances and a more harmonised approach made through the late 1970s into the early 1980s. The international fission track workshop, convened in Pisa, Italy in September 1980 and attended by over forty scientists, marked a turning point in method development and calibration as it confronted a number of persisting issues that needed to be resolved to enable the technique to become more widely accepted within the geochronological community. These included refinement of etching techniques to optimise track revelation and registration (Green and Durrani 1978), understanding the natural causes of track annealing (Burchart et al. 1979), neutron irradiation dosimetry and the value of the spontaneous-fission decay constant of ^{238}U (Hurford and Green 1981). The first inter laboratory comparison exercise was also conducted at this time to assess inter laboratory accuracy and precision (Naeser et al. 1981). As a consequence, most of the fission track papers published between 1970 and 1985 were concerned with methodological issues rather than application to geological problems. The few fission-track provenance studies published throughout the early to mid 1970's were concerned with providing a maximum age for the timing of deposition of unfossiliferous sandstones (Gleadow and Lovering 1974; McGoldrick, and Gleadow 1977) and stratigraphic ages of volcanic ashes (Naeser et al. 1974; Izett et al. 1974; Gleadow 1980; Johnson et al. 1982).

Many of the early volcanic ash studies were based on fission tracks in glass (e.g. Boellstorff and Steineck 1975) but when glass and zircon fission track ages were compared from the same sample it was realized that tracks were less stable in glass (Seward 1979) and thus like apatite it was not deemed suitable for provenance studies because in most cases source information would have been lost to thermal resetting. Thus when Hurford and Carter (1991) reviewed the few provenance-related papers published prior to 1990, zircon was the most widely used mineral due to its greater thermal stability. To a large extent this has not changed and apatite remained less widely used than zircon until the advent of double and triple dating approaches that combined analyses of fission track, U-Pb and (U-Th)/He on the same grains. Prior to this development few studies had attempted to use detrital apatites to constrain provenance. Notable exceptions were ocean drilling cores from locations where sedimentation rates were low and depths of burial shallow (Duddy et al. 1984; Corrigan and Crowley 1990; 1992; George and Hegarty 1995; Clift et al. 1996; 1997; 1998).

Working with zircon, the early provenance studies soon recognised the potential for data bias due to a range of factors that included small grains and counting areas, complex uranium zonation, or uncountable grains due to extremely high track densities, defects or inclusions. The need to sample the range of ages present in any given detrital sample is further complicated by variable etch rates between grains hence the need to make multiple grain mounts and apply different durations of etching (Naeser 1987; Garver 2003). Bias remains an issue as discussed more recently by Malusá et al. (2013) and in chapter 10.

Early provenance papers used simple histogram plots to identify mixed ages but it became apparent that the interpretation of detrital, mixed age datasets required more robust treatment in the form of statistical tools to display the observed fission track ages and to identify and extract the constituent component ages. The next section reviews the development of these statistical tools and interpretative strategies.

14.2. Development of interpretative strategies

Use of fission track thermochronometry to determine the provenance and stratigraphic age of sand and sandstones is contingent upon the detection of multiple age components and to be able to statistically estimate the most likely ages, and define the proportions and number of age components. For this reason unlike the population method that assumes an aliquot of apatite has the same uranium distribution and concentration as the aliquot used to count spontaneous tracks, the external detector method (EDM) is ideally suited to provenance studies because a fission track age is based on track counts from individual grains. The EDM is not prone to experimental bias from variations in uranium as is the case for the population method hence the variation within an EDM dataset is more likely to be natural rather than experimental.

During the early fission track studies it was not uncommon to count a relatively low (6 – 12) number of grains per sample. However, it was recognized that this failed to provide sufficient sampling to capture any natural variation and determine whether the grain ages belonged to a homogenous population, consistent with a Poisson distribution. A χ^2 test was used (Galbraith 1981) as a standard test for homogeneity, i.e. test if the individual data are consistent with a common ratio of spontaneous and induced counts. In many provenance studies a common outcome is evidence of over-dispersion with respect to Poisson variation in the spontaneous and induced track counts, which is consistent with a heterogeneous mix of grain ages. However, the χ^2 test can only indicate evidence for or against the null hypothesis. A low (< 0.05) ρ-value indicates heterogeneity within the dataset but it does not quantify the level of variation or show the nature of the distribution of the data. Realisation of the limitations of the χ^2 test led to the development of methods for graphical display of the grain data and probability models for quantifying the extent of variation and identification of component populations. These are described below.

Early provenance studies plotted single grain ages as histograms, but as this approach ignores the associated estimation errors it was realized that such plots cannot represent true age variation. To try and overcome this Hurford et al. (1984) and Kowallis et al. (1986) applied a Gaussian density function to each estimate to generate a continuous frequency curve or total probability density function. Whilst this appeared to account for grain ages with differing precisions by representing each estimate as a narrow density function the summary plot is an average density function that mixes both good and poor information (see Galbraith 1998). Hurford et al. (1984) even experimented with adjusting a smoothing factor to obtain greater resolution between overlapping peaks, a procedure that was further developed by Brandon (1996). However, Galbraith (1998) pointed out that since the probability density plots are not a kernel density estimate in the true sense such treatments are not valid, a point recently emphasized in the treatment of all types of detrital geochronological data by Vermeesch (2012).

To avoid problems associated with probability density plots Rex Galbraith devised the radial plot (Galbraith 1988; 1990) where the measurement error of each counted grain has a standard deviation on the Y scale. Radial plots are now widely used, including within the luminescence community who have devised a hybrid type of display known as the Abanico plot (Fig. 14.1) that combines both kernel density estimate and radial plot to display the distribution of ages of differing precision along with a picture of the age frequency distribution (Dietze et al. 2016). A number of software packages that uses these and other methods are freely available to users including radial/density plotter (Vermeesch 2009) and the Abanico R package (Dietze et al. 2016). Density plotter uses the statistical models for mixed fission track ages and minimum age (Galbraith and Green 1990; Galbraith and Laslett 1993), expanded upon in Galbraith (2005). These methods also underpin Binomfit, a windows program by Mark Brandon (1992) for the estimation of ages and uncertainties for concordant and mixed grain age distributions. The plotting program, PopShare (Dunkl 2002) employs a different mixture-modeling algorithm called the SIMPLEX method (Cserepes 1989) although it is unclear which function PopShare minimises. These methods all assume Gaussian distributions, which led to Sambridge and Compston (1994) devising a mixture modeling method that included non-Gaussian statistics to reduce the influence of outliers. Later, Jasra et al. (2006) developed Bayesian mixture models and used Markov chain Monte Carlo (MCMC) methods to fit the models, which is available as a standalone program written by Kerry Gallagher (BayesMix) or as an R package. Together these tools enable provenance studies to identify and extract components ages from populations of mixed ages.

14.3. Methodologies to enhance the interpretation of FT provenance data

From the earliest days of fission track stratigraphic and provenance studies it was recognized that interpretations of detrital datasets were strengthened by the inclusion of other types of geological data including zircon crystal morphology (Pupin 1980) and colour linked to α-radiation damage (Fielding 1970; Garver and Kamp 2002). Throughout the late 1980s and 1990s a succession of provenance papers were published exploring applications of detrital zircon fission track data tackling problems such as stratigraphic ages of biostratigraphically barren clastic sediments (Carter et al. 1995); dating weathered tuffs (Winkler et al. 1990) and identifying sources of basin sediments (and indirectly paleo sediment routing systems, Cerveny 1986; Garver and Brandon 1994; Carter et al. 1995) but interpretations often suffered from a lack of resolution to pinpoint sediment sources.

14.3.1 Zircon double and triple dating

A major stumbling block for studies of basin sediments sourced from large areas such as the foreland basins of the Andes or Himalaya is that regional exhumation from metamorphic temperatures often gave a non-unique zircon fission track age. This hindered identification of specific source locations within a mountain belt, and hence it was impossible to distinguish between volcanic and exhumational cooling ages. To overcome this Carter and Moss (1999) performed the first zircon double dating on Mesozoic fluvial sediments deposited in the Khorat Basin of eastern Thailand. Double dating is now an integral part of detrital studies in orogenic belts including the Himalayas (Carter et al. 2010; Najman et al. 2010), Pyrenees (Whitchurch et al., 2010), Andes (Thomson and Hervé 2002), Alaska, (Perry et al. 2009) as well as the Chinese Loess Plateau (Stevens et al. 2013). Rahl et al. 2003 adapted the approach by combining (U-Th)/He and U-Pb dating on zircons in a study of the Lower Jurassic Navajo Sandstone in Utah to constrain sediment routing, reycling and identify links to sources within the Appalachian orogeny. Campbell et al. (2005) applied the same approach to study the sources and level of recycling of sediment within the bedloads of the Ganges and Indus Rivers. Triple dating of single grains of zircon by combining fission track, (U-Th)/He and U-Pb was first reported by Reiners et al. (2004). See chapter 5 for further details.

One key issue when dealing with double datasets that include fission track data is that individual fission track ages are relatively imprecise compared to single grain U-Pb ages. Due to the large single grain uncertainties, the fission track age of a sample is based on a population (typically 20 to 30) of counts that should fall within a normal Poisson distribution. Within a Poisson distribution individual counts (grains) scatter around the true age and the amount of scatter can look significant when plotted against the comparatively precise U-Pb grain ages giving the impression that double data are bad. This is illustrated in Fig. 14.2 that compares zircon double dating results for the Tardree Rhyolite zircon (Ganerød et al. 2011), which is sometimes used as an internal fission track age standard. The fission track data have a central age that matches (within error) the true age and the population of counts all fall within a Poisson distribution, i.e. the data are not overdispersed. However, not all of the individual ages lie within error of the 1:1 line hence double dating interpretations should avoid low numbers of fission track grain ages and not make interpretations based on a single grain fission track age.

14.3.2. Apatite double and triple dating

Apatite can also be dated by the uranium-lead method (Thomson et al. 2012) and has been developed as a thermochronometer that can distinguish cooling paths over the interval between ~375 and 570 °C (Cochrane et al. 2014). Combination with fission track (double dating) provides a powerful tool for discerning provenance and a useful introduction to laser ablation ICPMS analyses for both fission track and U-Pb analysis can be found in Chew et al. (2011; 2012). For greater resolution of thermal history some studies have resorted to obtaining triple dates (FT, U-Pb and (U-Th)/He) from the same apatite grain. Carrapa et al. (2009) was the first to report triple dating in a study directed at understanding Andean mountain building in the Paleozoic and Cenozoic. In a study on detrital grains extracted form sedimentary rocks obtained from drillcore in the Victoria Land Basin of Antarctica, Zattin et al. (2012) applied triple dating on apatite (fission track, U-Pb and (U-Th)/He) to constrain a period of Oligocene exhumation in the Transantarctic Mountains and place constraints on basin evolution and provenance. U-Pb dating was performed on selected grains within the fission track mount and these were then removed for helium analyses.

14.4. Development of novel methods for detrital zircon provenance

Where next? The content of Ti in zircon is a function of crystallization conditions (Watson et al. 2006). Average, zircons from mafic igneous rocks have higher Ti concentrations than those from felsic rocks and therefore some have considered this as a potential tool for discrimination between sources of detrital zircon. However it has been found that mafic rocks can have values similar to felsic rocks and vice versa (Fu et al. 2008) and therefore the method cannot be used to blindly link a detrital zircon to a felsic or basic source. Further, the Ti-in-zircon thermometer is based on a calibration that assumes rutile as the Ti-buffering phase, which cannot be assumed in detrital systems. It can however, be used to discriminate sources when the source area is well described and in this regard it may prove a useful provenance tool as a complement to zircon fission track data along with (U-Th)/He and U-Pb ages.

Hafnium isotopes are increasingly measured on samples/grains with U-Pb ages, the goal being to determine if the zircon provenance was juvenile, evolved or mixed (Patchett et al. 1981). The Lu-Hf isotopic system can be combined with U-Pb dating on single grains to provide more robust constraints on crustal residence age as demonstrated for rocks in the Himalayas (Richards et al. 2005). Carter (2007) presented an example of hafnium isotope data measured on zircon FT and U-Pb dated grains from Himalayan sands in Bangladesh that demonstrated the potential of this combined approach for discerning sources within distinct tectonostratigraphic units. Although this example demonstrated the viability of measuring Hafnium on FT dated zircons as yet no study appears to have adopted this approach.

14.5. Development of novel methods for detrital apatite provenance

Heavy mineral associations, and geochemical variations have proven essential aids to provenance interpretations (Yim et al. 1985; Baldwin et al. 1986; Kowallis 1986; Garver and Brandon 1994; Lonergan and Johnson 1998; Ruiz et al. 2004). An interesting stratigraphic example used cathodoluminescence related to chemical concentrations and fission track densities in apatite as a tracer to correlate hydrothermal tin-tungsten veins in the Panasqueira mines of Portugal (Knutson et al. 1985). Because apatites contain wide variations in trace elements there is significant potential to exploit elemental variations to provide additional source information.

Much of our understanding of trace elements in apatites stems from studies of the compositional controls on fission track annealing and helium diffusion. Gleadow and Duddy (1981) first made a connection between over dispersed apatite grain ages and a possible compositional control, later confirmed by the studies of Green et al. (1985, 1986) on samples from the same drill-holes in the Otway Basin that revealed a correlation between apparent apatite FT ages and grain chlorine content. Although apatite chlorine content is important it rarely accounts for all of the observed variation in grain ages within an over-dispersed data set. The likely reason for this is that a wide range of elements (over half of the periodic table) may be substituted into the Ca, P or anion sites of apatite $Ca_{10}(PO_4)_6$ (F,Cl,OH) (Elliott 1994) thus other trace elements, if suitably abundant, may also influence the annealing sensitivity of fission tracks e.g. Mn, Sr and Fe (Ravenhurst et al. 1993; Burtner et al. 1994; Carlson et al. 1999), rare earth elements and SiO_2 (Carpéna 1998). Likewise composition also has a role in controlling helium diffusion (Mbongo Djimbi et al. 2015). From a provenance perspective variation in trace elements is a positive as it may provide a means with which to define the provenance and type of host rock.

Dill (1994) was one of the first to consider the utility of using apatite trace element signatures to determine the origin of detrital apatites in clastic rocks by investigating the origin of apatites in Permian and Triassic red-beds from southeast Germany. His study was inconclusive, in part due to the uncertain influences of diagenetic alteration.

A more detailed study by Belousova et al. (2001) examined the composition of over 700 apatites from a range of rock types. Results showed how different rock types have distinctive absolute and relative abundances of many trace elements (including rare-earth elements, Sr, Y, Mn, Th), and that chondrite-normalised trace-element patterns could be used to develop a discriminant tree to recognise the original host rock of an apatite. More recently Bruand et al. (2016) showed how trace element analysis of apatite inclusions within zircon and titanite provide useful constraints on magmatic history and source whole-rock chemistry of their host magmas.

14.5.1. Combined apatite fission track and trace element analyses

Whilst conventional provenance studies have started to use apatite chemistry comparatively little work has been done to combine apatite compositional information with thermochronometric data to define the original host rock, possibly because as yet there is no comprehensive database of apatite compositions (Morton and Yaxley 2007). Allen et al. (2007) in a study of the provenance of Paleogene rocks on the Andaman Islands plotted apatite chlorine content against uranium content (Fig. 14.3A) to show that apatites from an older rock unit (Hopetown Conglomerate, Mithakhari Group) were not the same as in a younger rock unit (Andaman Flysch) i.e. the two rocks did not share the same sources of apatite. To further discriminate source regions this study also compared apatite single grain Nd measured on samples from the Andaman Flysch Formation with sands from the Himalayas. Figure 14.3B shows the Andaman Flysch data plotted as εNd units (Yaxis) against $^{147}Sm/^{144}Nd$ (X-axis). A compilation of published bulk rock data (Henderson et al. 2010) showed that plotting the data in this way enables discrimination between sources within the Indian and Eurasian plates. It is clear from Fig. 14.3B that the Andaman Flysch apatites came from sources within Eurasia and not India.

The Sm/Nd ratios of apatites typically range from 0.2 to 0.5 (Belousova et al. 2002) and are similar to those of average continental crust (c. 0.2: Taylor and McLennan 1985). Foster and Carter (2007) developed a novel provenance technique that combined fission track and in situ Sm–Nd isotopic measurement of detrital apatites to constrain the source rocks. First applied on modern river sands in the Himalayas, it was possible to tie apatite fission track age populations to specific Himalayan tectonostratigraphic units that were being eroded at different rates (Carter and Foster 2009). The method works well for apatites in which a relatively short period has elapsed since they were last equilibrated with the whole rock.

There is clearly significant potential for increasing the breadth of compositional data from apatite grains used in detrital thermochronometric analyses to enhance provenance interpretations. Figure 14.4. demonstrates how existing methods that combine thermochronometry and composition data can be used to distinguish between a volcanic arc source, plutonic rocks and older metamorphic crust. The next step is to be able to determine the igneous rock type. In this regard apatite has significant potential as it is a relatively early-crystallizing phase and has a high partition coefficient for REE. Abundances of F, Mn, Sr and REE are known to vary with rock type (e.g. between peraluminous I-type and S-type rocks) and have been judged to be suitable provenance indicators (Belousova et al. 2002; Chu et al. 2009). Use of REE and trace-element patterns based on in situ measurement by LA-ICPMS and electron microprobe analysis to develop a discriminant tree of source rock types would seem the most obvious place to start and combined dating and trace element studies are likely to become more commonplace in the future.

14.6. Concluding remarks

This short review has outlined the methodological developments and interpretative strategies, built largely on the development of fission track analysis, that underpin detrital thermochronometry. Although fission track has been the principal thermochronometer used in provenance studies, with improved understanding of helium diffusion and the advent of in situ analyses wider use of detrital (U-Th)/He analyses should increase the fidelity of provenance interpretations. Whilst thermochronometry data are undeniably important, combining age data with geochemical information obtained from the same grains may further strengthen provenance studies interpretations. The latter remains relatively unexplored and there is considerable scope for the development of new methodologies.

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Figure Captions

Fig. 14.1. Example of an Abanico plot (Dietze et al. 2016) that combines the radial plot and kernel density plots in a single diagram.

Fig. 14.2. Zircon double dating of the Tardree Rhyolite. This volcanic rock has the same U-Pb and FT age that record the time of formation. The individual fission track data appear significantly scattered compared to the U-Pb ages. This scatter is consistent with a normal Poisson distribution that gives a fission track central age consistent with the true age.

Fig. 14.3. Compositions of apatites from the Paleogene Andaman Flysch, exposed on South Andaman Island are compared against apatite from a stratigraphically older unit (Hopetown conglomerate). Clasts in the Hopetown conglomerate show local arc sources. Plot A) compares apatite chlorine and uranium concentrations and shows two distinct sources. Low uranium chlorine rich grains are typical of volcanic sources. It has been suggested that the Andaman Flysch is Bengal Fan material eroded from the Himalayas. In B) apatite Nd compositions, plotted as **ε**Nd units against ^{147}Sm/^{144}Nd, belong to the Eurasian plate field and rule out Indian plate sources.

Fig. 14.4. Example of the application of single grain techniques coupled with fission-tracks in detrital studies. Fission-tracks analysis of a detrital sample collected from a river draining a young volcanic source (A) and granitic pluton (C) exhumed together with its gneissic country rock (B) only allows the discrimination of two age peaks. The differentiation of the relative contribution of (B) and (C) can be achieved by looking at the different apatite Sm/Nd and zircon Lu/Hf signatures in the two sources. Note that the relative contributions from (A), (B) and (C), as obtained by analysing different mineral species, differ due to non-homogeneous fertility of apatite and zircon in the source rocks (see Chap. 7).

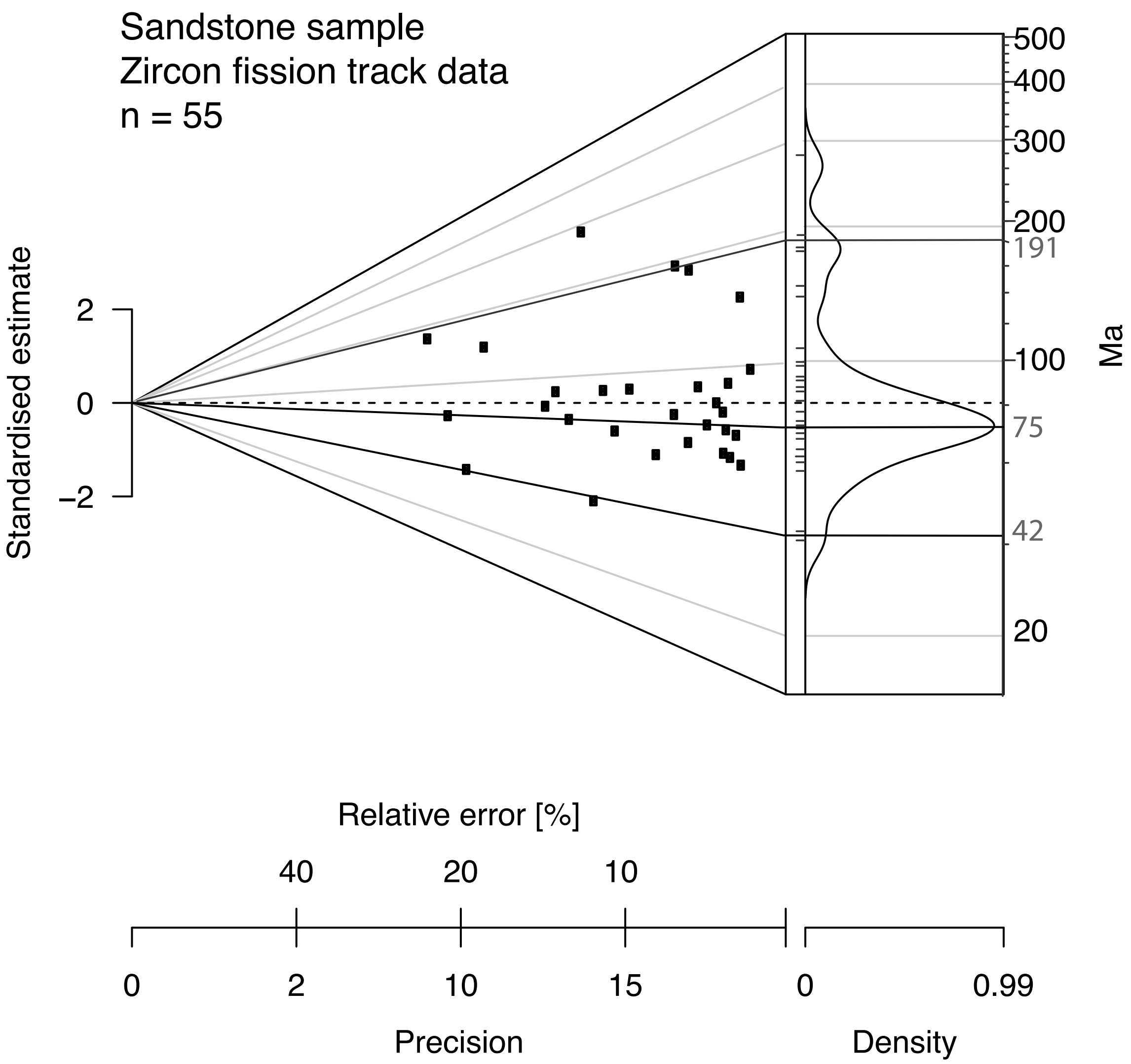

Sandstone sample
Zircon fission track data
n = 55
Standardised estimate
2
0
−2
500
400
300
200
191
100
75
42
20
Ma
Relative error [%]
40
20
10
0
2
10
15
0
0.99
Precision
Density

Fig. 14.1

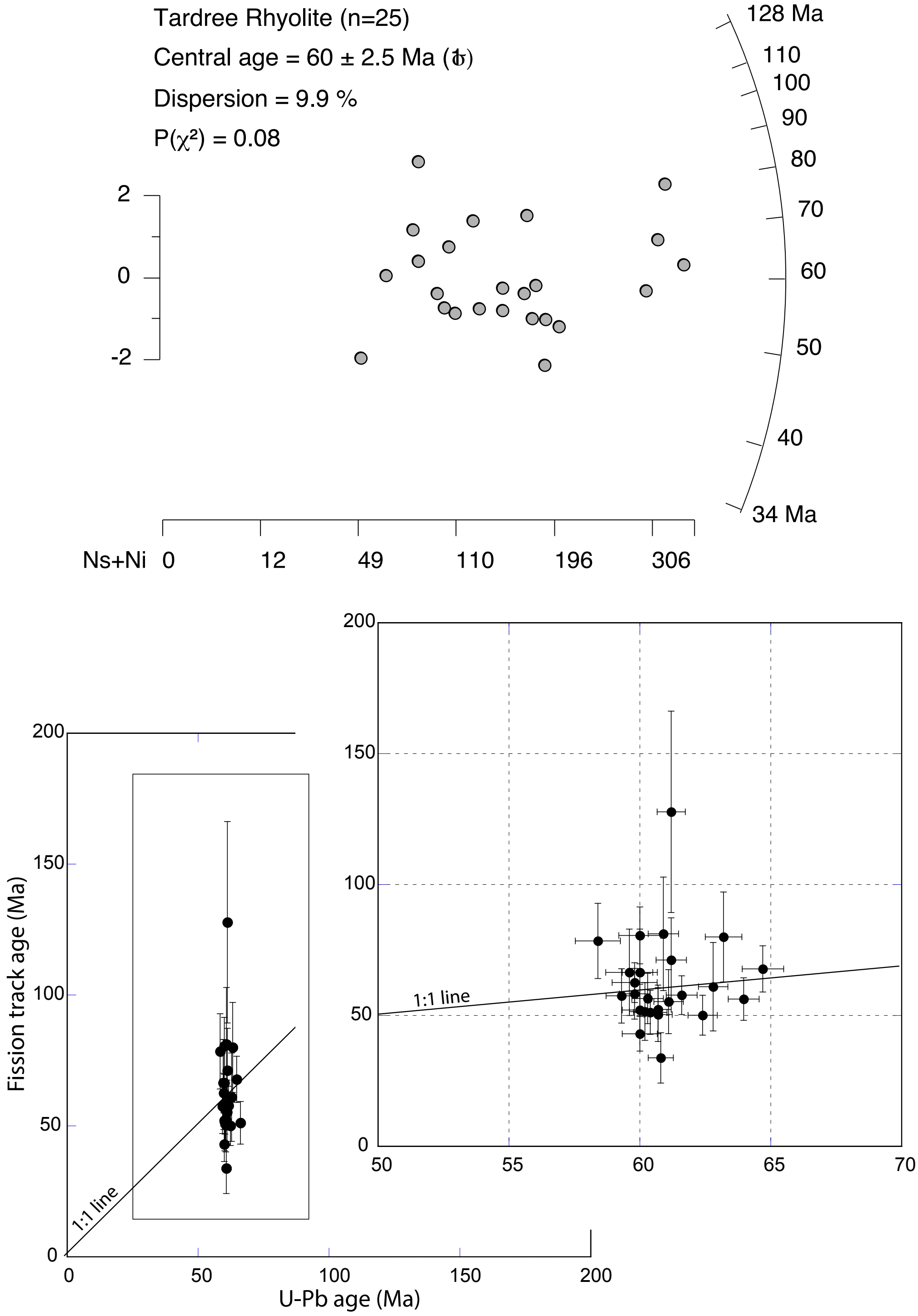
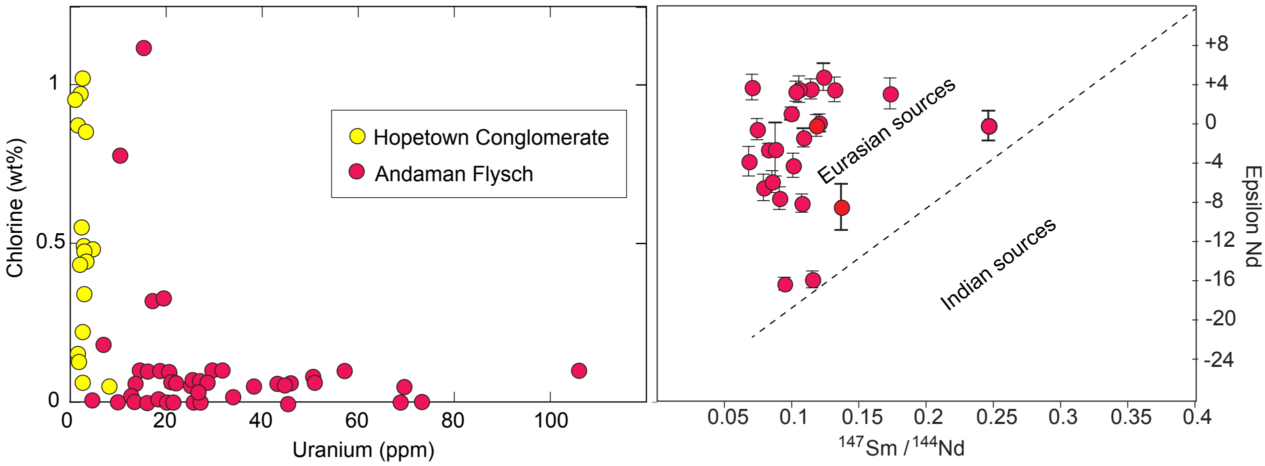
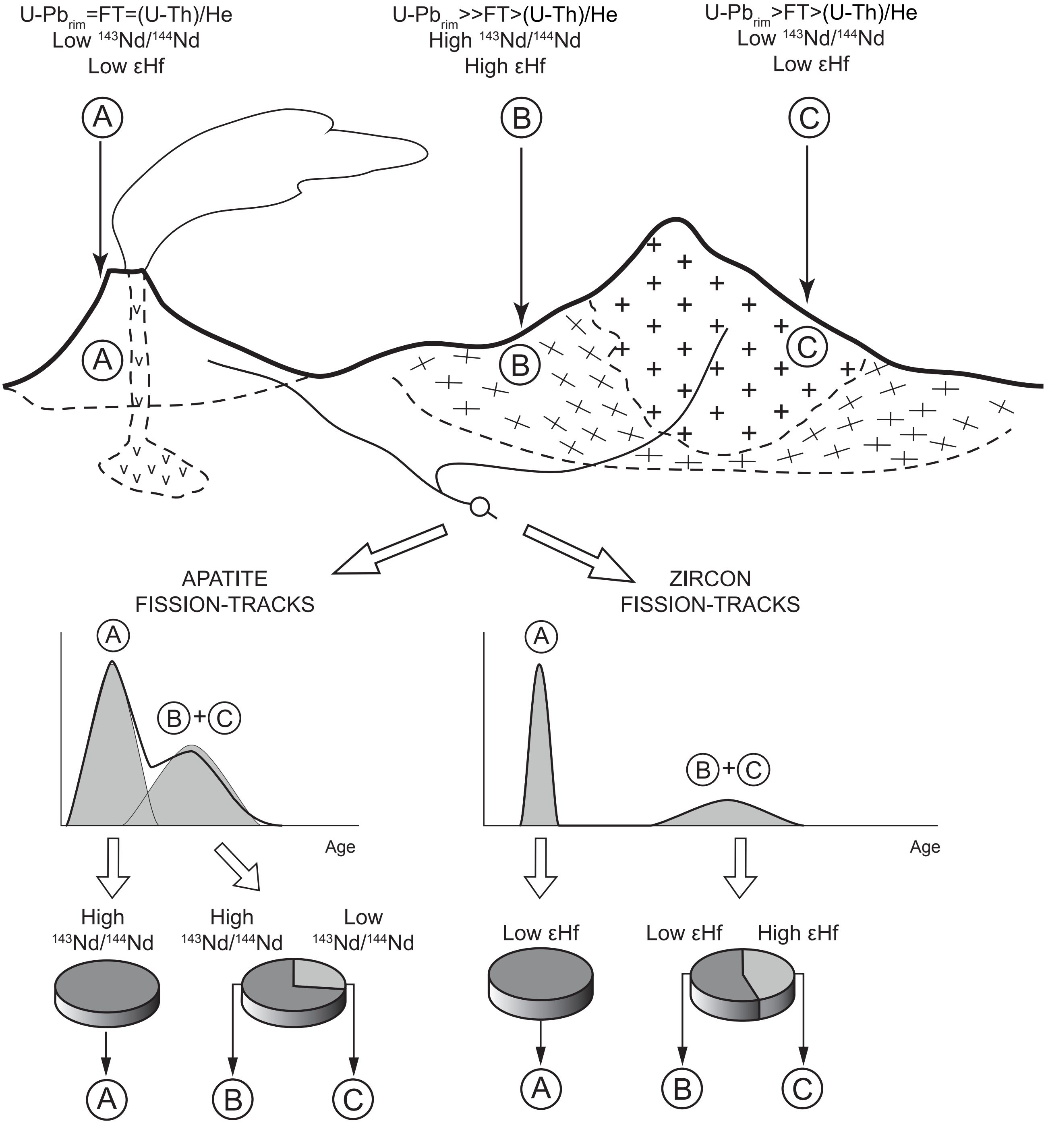

Tardree Rhyolite (n=25)
Central age = 60 ± 2.5 Ma (1σ)
Dispersion = 9.9 %
P(χ²) = 0.08
128 Ma
110
100
90
80
70
60
50
40
34 Ma
2
0
-2
Ns+Ni
0
12
49
110
196
306
Fission track age (Ma)
U-Pb age (Ma)
1:1 line
1:1 line

Fig. 14.2



Chlorine (wt%)
1
0.5
0
0
20
40
60
80
100
Uranium (ppm)
Hopetown Conglomerate
Andaman Flysch
Eurasian sources
Indian sources
+8
+4
0
-4
-8
-12
-16
-20
-24
Epsilon Nd
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4
147Sm / 144Nd

Fig. 14.3



U-Pb rim=FT=(U-Th)/He
Low 143Nd/144Nd
Low εHf
U-Pb rim>>FT>(U-Th)/He
High 143Nd/144Nd
High εHf
U-Pb rim>FT>(U-Th)/He
Low 143Nd/144Nd
Low εHf
A
B
C
APATITE
FISSION-TRACKS
ZIRCON
FISSION-TRACKS
B+C
Age
High
143Nd/144Nd
High
143Nd/144Nd
Low
143Nd/144Nd
Low εHf
Low εHf
High εHf

Fig. 14.4