5 Integration of Fission-Track Thermochronology with Other Geochronologic Methods on Single Crystals

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Abstract

Fission-track (FT) thermochronology can be integrated with the U–Pb and (U–Th)/He dating methods. All three radiometric dating methods can be applied to single crystals (hereafter referred to as "triple-dating"), allowing more complete and more precise thermal histories to be constrained from single grains. Such an approach is useful across a myriad of geological applications. Triple-dating has been successfully applied to zircon and apatite. However, other U-bearing minerals such as titanite and monazite, which are routinely dated by single methods, are also candidates for this approach. Several analytical procedures can be used to generate U–Pb—FT—(U–Th)/He age triples on single grains. The procedure introduced here combines FT dating by LA-ICPMS and in situ (U–Th)/He dating approach, whereby the U–Pb age is obtained as a by-product of U–Th analysis by LA-ICPMS. In this case, U–Pb, trace element and REE data can be collected simultaneously and used as annealing kinetics parameter or as provenance and petrogenetic indicators. This novel procedure avoids time-consuming irradiation in a nuclear reactor, reduces multiple sample handling steps and allows high sample throughput (predictably on the order of 100 triple-dated crystals in 2 weeks). These attributes and the increasing number of facilities capable of conducting triple-dating indicate that this approach may become more routine in the near future.

5.1 Introduction

The FT method is a powerful dating technique that can be used to constrain the timing and rates of a wide range of geological processes occurring in the uppermost kilometres of Earth's crust in the temperature range of ~60–350 °C. The major applications of the method include delineating the timing of rock exhumation (central for understanding the dynamics of orogenic systems and cratonic area), basin studies (revealing provenance of the material and burial/exhumation history), dating of volcanic eruptions, fault activity, genesis and preservation potential of economic mineralisations and many others (see Part II of this book, review books by Wagner and van den Haute (1992), Bernet and Spiegel (2004), Reiners and Ehlers (2005), Lisker et al. (2009), and some classic papers, e.g. (Wagner and Reimer 1972; Gleadow et al. 1983; Hurford 1986; Gleadow and Fitzgerald 1987; Green et al. 1989a, b; Gallagher et al. 1998; Ketcham et al. 1999; Kohn and Green 2002).

The FT method is based on the spontaneous fission of ^{238}U (Price and Walker 1963; Fleischer et al. 1975) in minerals like zircon, apatite and titanite (see Chap. 1 Hurford 2018). Spontaneous fission is only one of several decay mechanisms (e.g. U–Pb, (U–Th)/He, Lu/Hf, and Sm/Nd) that can be applied as geochronometers to these minerals and which provide complementary information on the cooling history. Until the late 1990s, the combined application of FT and other geochronometer(s) to the same crystals was not feasible due to technical limitations, although minerals from the same rock were often analysed using different techniques to constrain a time–temperature history. Thus in the majority of studies, the FT method was applied as a stand-alone technique focused solely on low-temperature geological processes. In the years to follow, technical and methodological advances paved the way for development of so-called in situ *multi-dating*. These include the ability to analyse smaller sample volumes, advances in in situ analytical techniques (e.g. laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS), ion microprobe dating by secondary ion mass spectrometry (SIMS) or sensitive high-resolution ion microprobe (SHRIMP) instruments), the introduction of a complementary FT dating methodology utilising LA-ICPMS (Cox et al. 2000; Svojtka and Košler 2002; Hasebe et al. 2004) and the emergence of

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the (U–Th)/He method as an additional and complementary low-temperature method (Zeitler et al. 1987; Farley 2002). In the in situ multi-dating approach, single minerals are analysed by FT method in combination with U–Pb or (U–Th)/He methods (hereafter termed *double-dating*; Carter and Moss 1999; Carter and Bristow 2003; Donelick et al. 2005; Chew and Donelick 2012) or by all three methods together (hereafter termed *triple-dating*; Reiners et al. 2004a; Carrapa et al. 2009; Danišík et al. 2010a; Zattin et al. 2012).

Multi-dating offers several advantages over the single method approach. For instance, it allows unprecedented, detailed reconstruction of thermal histories on single grains. These histories may cover the full spectrum of geological processes from crystal formation, through metamorphic overprint to final exhumation. This has an enormous application potential in Earth Sciences, in particular for detrital geochronology. However, this multi-dating approach on a single crystal has been only rarely applied and is still relatively new. For this reason, in this chapter a brief review of the history of multi-dating involving the FT method will be provided, along with a description of the rationale and theoretical background, and potential and limitations. Existing triple-dating analytical procedures will be described, and a brief introduction given to a new triple-dating approach that is currently being developed at Curtin University. The chapter will close with some applications and proposals for future directions.

5.2 Historical Perspective

Double-dating (FT and U–Pb methods applied to the same crystal) was first introduced by Carter and Moss (1999). These authors analysed detrital zircons from the Khorat Basin (Thailand) by FT using the external detector method (EDM, e.g. Gleadow 1981) to unravel the low-temperature thermotectonic evolution of the source terrains. Then, the same grains were U–Pb dated using SHRIMP to identify their crystallisation age. In addition to introducing the double-dating concept, this study highlighted that without complementary U–Pb data, the FT data alone would lead to misinterpretation of the source area and other erroneous conclusions (Carter and Moss 1999; Carter and Bristow 2000). Despite the demonstrated potential for provenance studies and exhumation studies (Chap. 14; Carter 2018; Chap. 15; Bernet 2018), the combined SIMS/SHRIMP U–Pb and EDM FT double-dating approach was subsequently used only twice (Carter and Bristow 2003; Bernet et al. 2006), likely because U–Pb dating by ion microprobe is a time-consuming and expensive technique, and more appropriate for other applications. Soon after, zircon U–Pb—(U–Th)/He double-dating (Rahl et al. 2003; Campbell et al. 2005; Reiners et al. 2005; McInnes et al. 2009; Evans et al. 2013) was also introduced, in which the zircon FT method is replaced by the (U–Th)/He thermochronometer that has a similar temperature sensitivity range and offers a less labour-intensive, higher sample throughput approach.

The renewed interest in U–Pb—FT double-dating started in the mid-2000s and was associated with the advent of LA-ICPMS into the field of thermochronology. First, a new methodology of FT dating employing LA-ICPMS to measure ^{238}U directly, replacing the conventional thermal neutron irradiation approach, was introduced (Cox et al. 2000; Svojtka and Košler 2002; Hasebe et al. 2004), dramatically increasing the speed of FT analysis and sample throughput. Soon after, LA-ICPMS methodology for FT dating of zircon was enhanced by adding the capability to determine the U–Pb age for each FT dated zircon grain by default (Donelick et al. 2005). In addition, the relatively recent introduction of new matrix-matched reference materials and new approaches to the common Pb correction allowed combined FT and U–Pb dating by LA-ICPMS to be routinely applied on apatite (Chew et al. 2011; Chew and Donelick 2012; Thomson et al. 2012). LA-ICPMS thus provided a more convenient, faster, less expensive but sufficiently precise and accurate means for routine U–Pb dating (e.g. Košler and Sylvester 2003). Nowadays, in FT studies using the LA-ICPMS approach, the provision of both FT and U–Pb ages on single grains is routine and an increased application of FT—U–Pb double-dating is noticeable in the literature (e.g. Shen et al. 2012; Liu et al. 2014; Moore et al. 2015).

At the time of writing, only one conference abstract and three research papers on triple-dating involving the FT method have been published (Reiners et al. 2004a; Carrapa et al. 2009; Danišík et al. 2010a; Zattin et al. 2012). The concept of zircon triple-dating was first introduced by Reiners et al. (2004a). The authors applied a combination of EDM FT, LA-ICPMS U–Pb and conventional (U–Th)/He dating to detrital zircon grains. Assuming that the measured ages record the time of cooling through the closure temperature (Dodson 1973; cf. Chap. 10; Malusà and Fitzgerald 2018a, b), they reconstructed cooling trajectories constrained by crystallisation ages, and cooling ages marking the passage through the ~240 and ~180 °C isotherms (i.e. nominal closure temperatures for zircon FT and zircon (U–Th)/He systems, respectively; Hurford 1986; Reiners et al. 2004b) for single zircon crystals. This demonstrated the potential of triple-dating to provide more information than double-dating approaches (both U–Pb—FT and U–Pb—(U–Th)/He). A similar concept utilising EDM FT, multi-collector LA-ICPMS (LA-MC-ICPMS) U–Pb and conventional (U–Th)/He dating was applied to detrital apatite by Carrapa et al. (2009) and Zattin et al. (2012). Both studies demonstrated the capability of this approach to obtain ages that were interpreted by the authors to represent cooling through the ~500, ~110 and ~65 °C isotherms (i.e. nominal closure temperatures for

U–Pb, FT and (U–Th)/He systems in apatite, respectively; Cherniak et al. 1991; Wagner and van den Haute 1992; Farley 2000). Using the triple-ages, the authors elucidate the provenance of the grains and their thermal history at higher temporal and spatial resolution than would be possible by using single-dating methods. An alternative approach to, and application of, apatite triple-dating was presented by Danišík et al. (2010a) who applied ID-TIMS U–Pb, EDM FT and conventional (U–Th)/He dating to a hydrothermal apatite aggregate in an attempt to constrain the thermotectonic evolution of basement rocks and investigate apparent discrepancies between FT and (U–Th)/He data obtained by single methods.

Currently, the major obstacle of the triple-dating approaches described above relates to the relatively complicated, time-consuming, labour-intensive analytical procedures with multiple handling steps and to accessibility of the analytical instruments. There are only a few institutions pursuing research directions both in high-temperature and low-temperature geochronology and which are accordingly equipped with a FT laboratory, and LA-ICPMS and noble gas mass spectrometer instruments. However, recent progress in the automation of FT counting and offsite data processing (Gleadow et al. 2009), together with increasing numbers of accessible LA-ICPMS laboratories and the development of fast throughput in situ (U–Th)/He dating techniques (Boyce et al. 2006; van Soest et al. 2011; Vermeesch et al. 2012; Tripathy-Lang et al. 2013; Evans et al. 2015; Horne et al. 2016) may help to overcome at least some of these limitations and holds great promise for triple-dating in the future.

5.3 Rationale of Multi-dating

The rationale of the double- and triple-dating approaches is in the combined application of dating methods with different temperature sensitivities to the same crystals, which enables geoscientists to extract a more complete and more detailed picture of the cooling history. Principles of the radiometric dating techniques complementing FT in double- and triple-dating approaches have been described in several comprehensive review books and papers (e.g. Farley 2002; Reiners 2005 for (U–Th)/He dating; Hanchar and Hoskin 2003; Schaltegger et al. 2015 for U–Pb dating), and therefore are not detailed here. In brief, the FT method is based on the accumulation (and annealing) of linear damage (fission tracks) produced by spontaneous fission of ^{238}U; the U–Pb method is based on the accumulation of Pb produced by a series of alpha and beta decays of U; and the (U–Th)/He method is based on the accumulation of ^{4}He produced by alpha decay of U, Th and Sm.

Temperature sensitivity ranges for the U–Pb, FT and (U–Th)/He systems in most common mineral phases suitable for double- and triple-dating are illustrated in Fig. 5.1. In general, the U–Pb system is sensitive to higher temperatures (i.e. 350–1000 °C) than the FT and (U–Th)/He systems and typically records process occurring in upper-mantle to mid-crustal levels (e.g. Chew et al. 2011; Cochrane et al. 2014; Schaltegger et al. 2015). FT and (U–Th)/He systems, in contrast, are sensitive to lower temperatures (40–350 °C) and typically record upper crustal processes (e.g. Ehlers and Farley 2003; Danišík et al. 2007; Malusà et al. 2016).

It should be noted that the concept of applying a range of methods to different aliquots of the same mineral from the same rock is not new (see, e.g., McInnes et al. 2005; Vermeesch et al. 2006; Siebel et al. 2009). So, the question to be asked is—what is the advantage of combining FT dating with U–Pb and/or (U–Th)/He methods and applying these to a single crystal? To answer this question, it is worthwhile to consider the strengths and limitations of the FT method and appreciate possible ambiguity in some FT data. Even though U–Pb and (U–Th)/He methods have also strengths and limitations to be aware of that discussion is beyond the scope of this chapter.

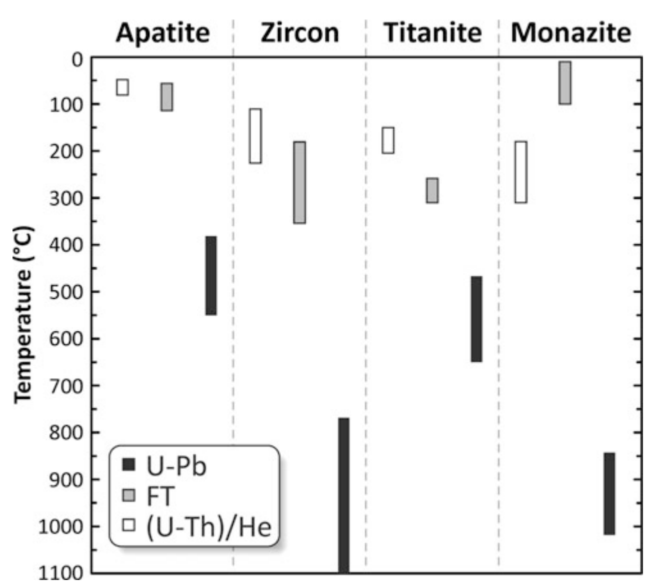

Fig. 5.1 Characteristic temperature sensitivity ranges for U–Pb, FT and (U–Th)/He radiometric systems in most common minerals suitable for triple-dating. Partial annealing/retention zones were calculated with closure software (Brandon et al. 1998) using the data for apatite after Cherniak et al. (1991), Ketcham et al. (1999), Chamberlain and Bowring (2001), Farley (2000); for zircon after Brandon et al. (1998), Cherniak and Watson (2001, 2003), Cherniak (2010), Rahn et al. (2004), Guenthner et al. (2013); for titanite after Cherniak (1993, 2010), Coyle and Wagner (1998), Hawkins and Bowring (1999), Reiners and Farley (1999); for monazite after Cherniak et al. (2004), Gardés et al. (2006), Boyce et al. (2005), Weise et al. (2009)

The major strength of the FT method is in its ability to discriminate not only the timing of cooling, but also the style of cooling within the partial annealing zone (PAZ), as recorded by the track length distribution (e.g. Gleadow et al. 1986a). FT age and track length data in apatite often enable robust reconstructions of best-fit time–temperature envelopes via forward and/or inverse modelling (Ketcham 2005; Gallagher 2012), i.e. whether they record a distinct geologic event or they are the result of a more complex path (see Chap. 3; Ketcham 2018; Chap. 8; Malusà and Fitzgerald 2018a, b). Despite the possibility of Californium irradiation technique that allows etchant to reach confined tracks (Donelick and Miller 1991; Chap. 2; Kohn et al. 2018), measurement of statistically robust track length distributions (typically 100 track length per sample or per significant age population) may not always be feasible, in particular in samples with young (Late Cenozoic) FT ages and/or low uranium concentration. A poorly defined confined track length distribution makes it difficult to interpret non-reset detrital samples that have multiple age populations. Whether track length data can be obtained or not, the addition of U–Pb and/or (U–Th)/He ages provides additional higher- and lower-temperature constraints to cooling trajectories that can greatly improve the understanding and interpretation of FT ages. When track length data are available, the addition of U–Pb and/or (U–Th)/He data can still greatly benefit the interpretation by permitting more accurate reconstruction of the time–temperature history, whereby U–Pb age constrains the high-temperature part of the cooling trajectory and (U–Th)/He data provide additional constraints for low-temperature processes (e.g. Stockli 2005; Green et al. 2006; Emmel et al. 2007).

Because of the relatively low number of fission tracks counted in each grain, the FT method yields relatively low precision on single-grain ages. For example, standard errors in young samples are commonly >20% at 1σ compared with <2 and 2–5% errors for the U–Pb by LA-ICPMS and conventional (U–Th)/He methods, respectively. In addition, dispersion of single-grain FT ages is common, even in apatites from rapidly cooled rocks. For example, a typical range of 25–50 single shard/grain EDM FT ages obtained on age standards regularly measured for zeta-calibration is commonly from $\sim$17 to $\sim$55 Ma (on Durango apatite) and from $\sim$15 to $\sim$50 Ma (on Fish Canyon zircons) (M. Danišík, unpublished data). Dispersion is due to the relatively few numbers of tracks counted as mentioned above, as well as compositional and structural variation of individual crystals, and thermal evolution in which slow or complex cooling through or into and out of the PAZ causes increased scatter of single-grain ages (e.g. Gleadow et al. 1986b). In the FT applications where single-grain FT ages are expected to form single age population (e.g. quickly cooled igneous rocks or quickly cooled, fully reset sediments), these problems are mitigated by analysing >20 crystals and calculating a population geometric mean age (a.k.a. *central age*, see Chap. 6; Vermeesch 2018) with corresponding standard error, which is commonly $\sim$3–5% at 1σ (Galbraith and Laslett 1993). However, in applications such as detrital dating studies, where crystals are derived from multiple sources and commonly produce complex FT age distributions, dispersion of single-grain FT ages will be greater. In detrital studies, confined track length distributions are typically not representative of the collective cooling history, because of different grain populations from different provenances, thus making interpretation of data sets challenging. Complementing FT ages with U–Pb and/or (U–Th)/He ages from the same grains is, therefore, invaluable in this respect, and triple-dating offers several advantages to overcome these issues:

- First, the combination of three ages allows a direct internal data quality check, where ages for apatite, zircon and titanite should follow the general trend of U–Pb age $\geq$ FT age $\geq$ (U–Th)/He age (e.g. Hendriks 2003; Lorencak 2003; Belton et al. 2004; Hendriks and Redfield 2005; Green et al. 2006; Ksienzyk et al. 2014), as dictated by the closure temperature concept (Dodson 1973), although there are exceptions. For example in old, slowly cooled terrains, an apatite FT age may be < (U–Th)/He age, as discussed in Chap. 21 (Kohn and Gleadow 2018). The multi-method approach therefore allows identification of analytical outliers (improving data set robustness), and of contaminant, diagenetic or authigenic grains that may present important geological information but that would not be otherwise detected using the FT method alone.
- Second, a lack of geological context can hamper accurate interpretation of single ages whereas multiple ages on the same crystal may mitigate this. For instance, without the prior knowledge of U–Pb and FT data, it is not possible to discriminate between “apparent” (U–Th)/He ages resulting from complex thermal histories causing partial resetting of the (U–Th)/He system and “cooling” (U–Th)/He ages resulting from simple cooling paths where the closure temperature concept applies (e.g. Stockli et al. 2000; Danišík et al. 2015).
- Third, the combination of the three ages may better constrain the cooling trajectories for single crystals—from crystallisation (or high-grade metamorphism) to final cooling. Detailed cooling histories may provide diagnostic fingerprints of the source terrain, allowing a more robust interpretation of detrital data than could be achieved by using one method alone.

Finally, it may be argued that single-grain multi-dating, notably for detrital studies should be pursued because it is technologically possible and markedly more efficient. When compared to traditional FT dating protocols, modern LA-ICPMS U–Pb and (U–Th)/He dating procedures are largely automatised and do not require permanent attendance of an operator. Given recent technological and methodological innovations (e.g. FT dating by using LA-ICPMS providing U–Pb dating as a by-product (Donelick et al. 2005; Chew and Donelick 2012), the automation of FT counting (Gleadow et al. 2012), developments of high-throughput in situ U–Pb and (U–Th)/He dating techniques (Boyce et al. 2006, 2009; van Soest et al. 2011; Vermeesch et al. 2012; Tripathy-Lang et al. 2013; Evans et al. 2015) and the improved accessibility of the required instruments (e.g. growing number of LA-ICPMS, FT and He laboratories, the possibility of remotely controlled analytical measurements, offsite data reduction, etc.), the acquisition of double- and triple-age data on single crystals are now practical and may soon become the norm.

5.4 Analytical Procedures for Combined FT, U–Pb and (U–Th)/He Dating

Analytical protocols for triple-dating using combined U–Pb, FT and (U–Th)/He methods involve measurements of parent nuclides (U, Th, ±Sm) and their daughter products (Pb isotopes, spontaneous fission tracks and He, respectively) in the same crystals. Published and newly proposed workflows for triple-dating are summarised in Fig. 5.2.

Selection of the analytical procedure depends on several factors such as size and quantity of minerals, accessibility of analytical instruments, time available for analytical work and desired data quality with regard to precision and accuracy. Large, (>∼2 mm) single crystals or crystal aggregate(s) (with identical magmatic and cooling history) can be crushed or disaggregated in a mortar in order to obtain small shards (preferably >50 μm), and these can be dated separately using fully destructive (e.g. ID-TIMS U–Pb, conventional (U–Th)/He dating) or semi-destructive methods (U–Pb by SIMS or LA-(MC)-ICPMS, in situ (U–Th)/He, FT dating by EDM or LA-ICPMS). The main advantage of this approach is in the possibility of obtaining high-precision U–Pb data when using ID-TIMS (Parrish and Noble 2003) and more accurate and precise conventional (U–Th)/He ages by eliminating the need for alpha ejection correction (Farley et al. 1996) when analysing shards from grain interiors or applying mechanical abrasion removing the outer ∼20 μm of grain surface (Krogh 1982; Danišík et al. 2008). The primary limitation of this approach is that large U-bearing crystals suitable for triple-dating are rare in the nature. An example of the combined TIMS U–Pb, EDM FT and conventional (U–Th)/He dating of a cm-size, hydrothermal apatite can be found in Danišík et al. (2010a).

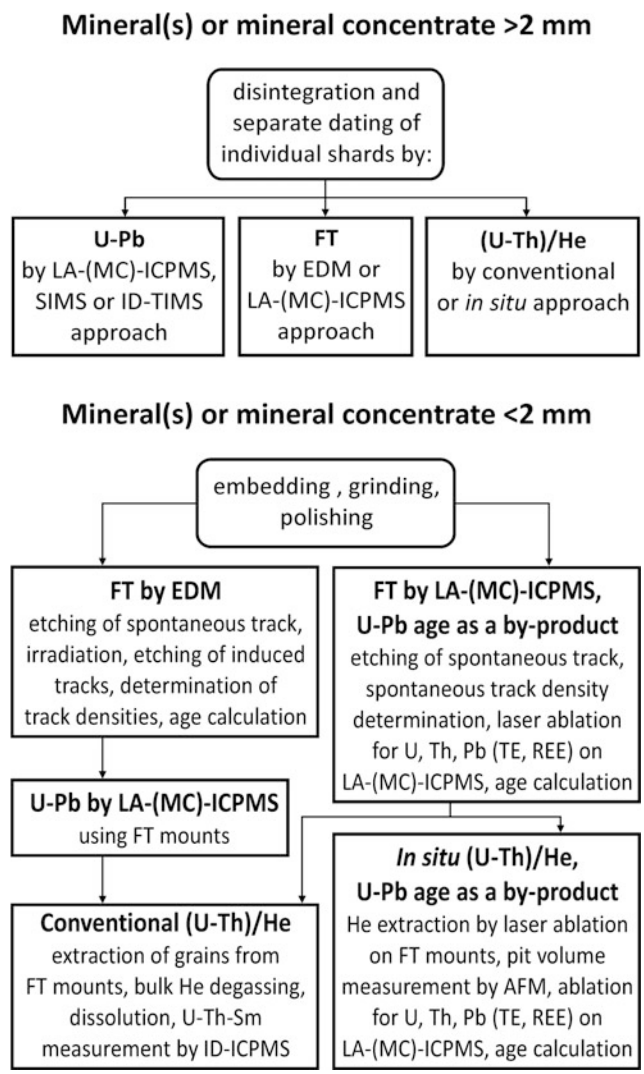

Fig. 5.2 Flow charts summarising feasible methodologies for triple-dating

Accessory heavy minerals that are typically found in the <250 μm size fraction need to be analysed using semi-destructive techniques during intermediate dating stages. The three studies reporting triple-dating of apatite (Carrapa et al. 2009; Zattin et al. 2012) and zircon (Reiners et al. 2004a), followed an almost identical protocol in which crystals were first dated by conventional EDM FT methods involving embedding, grinding, polishing, etching of spontaneous fission tracks, irradiation in a nuclear reactor, etching of induced tracks in the mica external detector and track counting allowing the age calculation. Then, the FT dated grains were dated by U–Pb method using LA-(MC)-ICPMS, and finally, the FT + U–Pb dated grains were extracted from the FT mounts and dated by a conventional (U–Th)/He method that involved determination of bulk He content using noble gas mass spectrometry, dissolution of crystals in acids and U–Th analysis by solution isotope dilution ICPMS

(Reiners et al. 2004a; Carrapa et al. 2009; Zattin et al. 2012). This approach suits detrital crystals of common size (typically >50 µm) and has the added advantage of providing the opportunity to simultaneously collect geochemical data during LA-(MC)-ICPMS analysis (e.g. Cl, F, trace elements, REE or Hf isotopes). These data can be very useful for characterising annealing kinetics in apatite for thermal modelling purposes (Barbarand et al. 2003; Ketcham et al. 2007a, b), as indicators of source rock lithology (e.g. trace elements in apatite; Morton and Yaxley 2007; Malusà et al. 2017), as petrogenetic tracers (e.g. REE in zircon; Schoene et al. 2010; Jennings et al. 2011), or as tracers of host rock origin (Hf in zircon; Kinny and Maas 2003; Flowerdew et al. 2007). The major limitations of this approach relate to sample irradiation in a nuclear reactor, which in some cases can be time-consuming, and in certain countries can sometimes take up to three months. There are also more sample handling steps when dealing with irradiated samples and then there is also a risk of crystal loss during the extraction from the FT mount (crystals are typically plucked out from the FT mount submerged in ethanol by using a sharp needle or tweezers with sharp ends) and loading into micro-tubes prior to He analysis. In addition, correction for the high common Pb content in young, low-U apatites can be difficult when using a quadrupole ICPMS for U–Pb dating (Chew et al. 2011; Thomson et al. 2012).

Some of these issues can be circumvented using a new triple-dating approach that is currently being developed in the GeoHistory Facility at the John de Laeter Centre (Curtin University). This approach combines FT dating by LA-ICPMS (Hasebe et al. 2004; Donelick et al. 2005; Chew and Donelick 2012) and in situ (U–Th)/He dating (Evans et al. 2015), with the LA-(MC)-ICPMS U–Pb age obtained as a by-product of either of the two methods. Preliminary results obtained on Durango apatite (Fig. 5.3) are in excellent agreement with the expected value, which holds a great promise for the future. This new approach can be briefly described as follows:

Crystals of interest (both apatite and zircon) are embedded into Teflon (DuPont PFA, Type 6000LP) as it, unlike epoxy, does not excessively degas and allows the desired pressure in the UHV cell to be obtained. Crystals are then ground to 4π geometry and sequentially polished using 9-, 3-, 1-µm-diamond and 0.3-µm-colloidal silica suspensions. After a thorough cleaning of the mounts, spontaneous fission tracks in polished crystals are revealed by etching using standard etching protocols. Spontaneous track density (i.e. number of tracks per known area), confined track lengths and Dpar values (Burtner et al. 1994) are then measured for selected grains under an optical microscope equipped with a high-resolution camera. The concentration of ^{238}U in the grains is then directly determined by LA-(MC)-ICPMS (Agilent 7700 s or NU Plasma II ICPMS both connected to

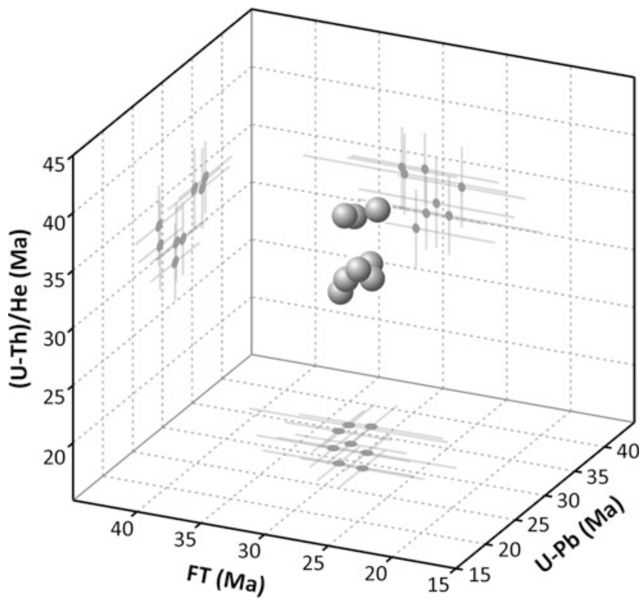

Fig. 5.3 3D scatter plot showing initial results of the newly proposed triple-dating methodology applied to shards of Durango apatite age standard (Ar–Ar reference age: 31.44 ± 0.18 Ma (2σ); McDowell et al. 2005). The procedure employed FT dating by LA-ICPMS (Chew and Donelick 2012) and in situ (U–Th)/He dating (Evans et al. 2015); spontaneous fission tracks were counted under a Zeiss Axioskop 2 microscope; isotopic data were collected on quadrupole noble gas mass spectrometer (Pfeiffer PrismaPlus) and an Agilent 7700 s single quadrupole ICPMS both connected to 193 nm ArF RESOlution COMPexPro 102 excimer laser. U–Pb ages are ^{207}Pb corrected ^{238}U/^{206}Pb ages calculated as Tera–Wasserburg concordia lower-intercept ages anchored through common Pb (after Stacy and Kramer 1975); FT ages were calculated using the scheme presented by Donelick et al. (2005) and Chew and Donelick (2012); in situ (U–Th)/He ages were calculated by using the first principle approach (Boyce et al. 2006). Uncertainties on U–Pb, FT and (U–Th)/He ages are reported at 1σ level

193 nm ArF RESOlution COMPexPro 102 excimer laser with S155 Laurin Technic laser ablation flow-through cell) and the FT age for each grain is determined following the protocols described by Hasebe et al. (2004), Donelick et al. (2005) and Chew and Donelick (2012). In addition to ^{238}U, the LA-(MC)-ICPMS permits measurement of Pb content (as well as a range of trace elements and REE, if desired), permitting calculation of a U–Pb age as a by-product (Donelick et al. 2005; Chew and Donelick 2012). Almost non-destructive FT analysis by LA-ICPMS utilising ~23 to 50 µm circular laser spots preserves enough space on the polished crystal surfaces for in situ (U–Th)/He analysis. Analytical procedures for in situ (U–Th)/He dating follow the protocols of Evans et al. (2015). The Teflon mount with target crystals is loaded into the UHV cell of the RESOchron instrument, and He is extracted from intact polished surfaces by laser ablation (using 33 or 50 µm spots). He content is determined on a quadrupole mass spectrometer (Pfeiffer PrismaPlusTM) by isotope dilution using a known volume of

^{3}He spike. The mount is then retrieved from the UHV cell, and the volume of the He ablation pits is measured using an atomic force microscope (although a confocal laser scanning microscope could also be employed; Boyce et al. 2006). This allows the He concentration to be determined which is required for the in situ (U–Th)/He age calculation (Boyce et al. 2006; Vermeesch et al. 2012). The mount is then reloaded into the flow-through cell (S155) for a third ablation to determine U, Th and Sm contents by LA-(MC)-ICPMS, allowing the final calculation of (U–Th)/He age. Similar to the first laser ablation for FT dating, this stage of LA-(MC)-ICPMS analysis permits determination of the U–Pb age as by-product (Boyce et al. 2006; Vermeesch et al. 2012; Evans et al. 2015).

The major advantages of such approach are in the significantly shorter analytical time and higher sample throughput achieved by an overall simplification of sample handling procedures for what is essentially three methods. There is no need to extract dated grains from the FT mounts and load them into micro-tubes for conventional He extraction, most of the analytical steps are automated and samples do not require irradiation for FT analyses. A realistic workflow suggests that ~100 crystals may be triple-dated in 2 weeks. Further advantages include the ability to date "problematic" crystals, not suitable for conventional (U–Th)/He analysis (e.g. crystals with extreme zonation of parent nuclides, structural inhomogeneities caused by radiation damage, mineral and/or fluid inclusions), and circumvention of an alpha ejection correction both achieved by targeting grain interiors (Boyce et al. 2006), and improved worker safety is that there is no need for grain dissolution for (U–Th)/He dating, thereby avoiding the use of hydrofluoric, nitric or perchloric acids, and there is no need for irradiation and training of workers in use of radioactive samples. However, at least four limitations to this approach should be considered:

- First, although FT dating by LA-ICPMS provides higher precision on relative uranium concentrations compared to the conventional EDM method (Donelick et al. 2006), this approach may not be suitable for strongly zoned crystals (Hasebe et al. 2004; Donelick et al. 2005).
- Second, the precision and accuracy of in situ (U–Th)/He ages may not be as good as conventional (U–Th)/He ages (Horne et al. 2016) due to the simplified assumption of the homogeneity in distribution of parent nuclides in dated minerals, inherited lower analytical precision of LA-ICPMS data in comparison to isotope dilution ICPMS data, and an additional source of uncertainty related to the pit volume measurements.
- Third, as in the previous EDM FT + LA-ICPMS U–Pb + conventional (U–Th)/He approach, triple-dating of young, low-U apatite can be problematic due to the high amount of common Pb and low abundance of radiogenic Pb and He.
- Fourth, currently there are only four laboratories publishing in situ (U–Th)/He data so the accessibility to this methodology is currently limited.

However, the advantages of triple-dating using this approach outweigh the limitations for many applications, such as, a case where large numbers of grains need to be analysed. In the future, it is anticipated that the number of laboratories with similar in situ capabilities will increase; thus, triple-dating is likely to become much more widely used in coming years.

The effect of chemical etching on (U–Th)/He systematics One of the critical requirements for successful triple-dating is that all radiometric decay schemes used are undisturbed during the multiple analytical steps. While sample embedding, grinding, polishing, FT counting and "cold" ablation by excimer laser should not alter the parent–daughter systems, the effect of chemical etching (required to reveal spontaneous fission tracks) on (U–Th)/He systematics may be a concern. The etching of apatite, titanite and monazite is safe in this regard as it is carried out at temperatures well below the temperature sensitivity of the (U–Th)/He system in these minerals. Routine procedures include etching in 5 or 5.5 M HNO_3 solution at 21 °C for 20 s for apatite (e.g. Donelick et al. 1999), HF–HNO_3–HCl–H_2O solution at 23 °C for 6–30 min for titanite (e.g. Gleadow and Lovering 1974) and in boiling (~50 °C) 37% HCl for 45 min for monazite (Fayon 2011). However, the effect of long and aggressive etching of fission tracks in zircon (10–100 h at 215–230 °C in a eutectic KOH–NaOH mixture; Zaun and Wagner 1985; Garver 2003; Bernet and Garver 2005) on He diffusion may be of concern as the etching temperatures are above the lower limit of the zircon He partial retention zone (~150 °C; Guenthner et al. 2013). To test this hypothesis, an experiment was conducted in which 15 zircon crystals from the Fish Canyon Tuff were etched in a eutectic KOH–NaOH mixture at 215 °C for 100 h, at which point the spontaneous fission tracks were revealed in all crystals. The etched crystals were then analysed, together with 15 unetched Fish Canyon Tuff zircons by conventional (U–Th)/He method. Comparison of the results (Fig. 5.4) shows no significant difference in (U–Th)/He ages of etched and unetched crystals, suggesting that the long and aggressive etching of spontaneous fission tracks in zircon is not an issue for subsequent (U–Th)/He dating and the triple-dating approach.

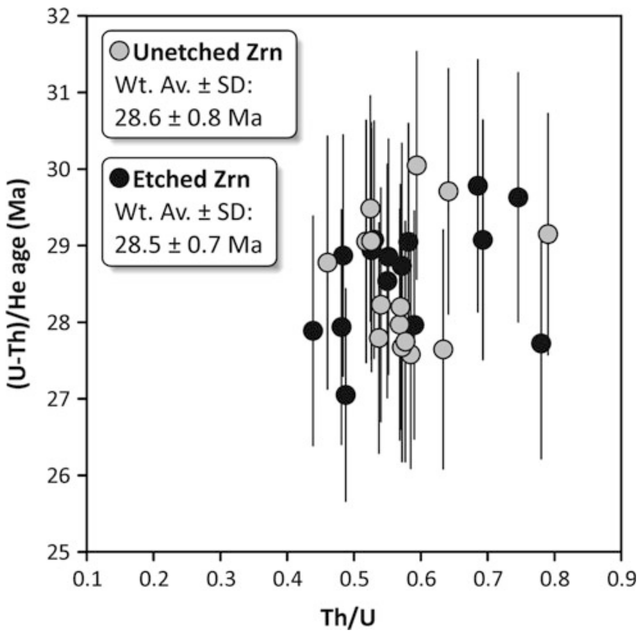

Fig. 5.4 Comparison of conventional (U–Th)/He ages obtained on chemically untreated and etched zircon crystals from Fish Canyon Tuff (reference (U–Th)/He age: 28.3 ± 1.3 Ma; Reiners 2005). Weighted averages of unetched and etched zircon are similar, demonstrating no significant effect of etching on (U–Th)/He system in the investigated sample. Etching conditions uses: NaOH–KOH eutectic melt, 215 °C, 100 h (Zaun and Wagner 1985; Garver 2003; Bernet and Garver 2005)

5.5 Applications

Triple-dating of single crystals yields an unprecedented amount of chronological information and is ideally suited to detrital dating studies (e.g. Carrapa 2010) where a large number of crystals need to be analysed in order to identify statistically significant age components with confidence (e.g. Vermeesch 2004). Depending on the dated mineral, single crystal triple-dating can resolve the magmatic, metamorphic and exhumation history of source terrains, establish maximum depositional ages and detect post-depositional heating events. Hypothetically, the combination of (tectono-)thermal events experienced by detrital grains should result in a unique combination of U–Pb, FT and (U–Th)/He ages for each grain (Fig. 5.5).

As discussed above, triple-dating provides significant advantages over single-dating methods in several aspects. The studies of Carrapa et al. (2009) and Zattin et al. (2012) show that triple-ages obtained on a relatively low number of grains allowed to derive more geological conclusions than those resolvable from 100 grains (the commonly adopted number in detrital studies) dated by a single method. Evaluation of the robustness of triple-dating with regard to identifying age components is beyond the scope of this study and needs to be further tested in future. However, it is likely that fewer grains will provide at least as much (and likely more) detailed provenance information than single-dating method approaches, applied to a high number of grains.

In addition to its application to zircon and apatite, triple-dating should be applicable to other common detrital minerals such as titanite and monazite that are (with some limitations) datable by U–Pb, FT and (U–Th)/He methods (e.g. Reiners and Farley 1999; Stockli and Farley 2004; Boyce et al. 2005; Siebel et al. 2009; Fayon 2011; Weisheit et al. 2014; Kirkland et al. 2016a, b). In addition to different closure temperatures for given radiometric systems (Fig. 5.1), each of these minerals may be representative of different source lithologies and also have different mechanical and chemical properties that translate to different stability during sedimentary transport (Chap. 7; Malusà and Garzanti 2018). Triple-dating applied to different minerals can therefore potentially enable more reliable identification of source areas and reconstruction of their individual thermal histories and can provide critical information on thermal events during different stages of the mineral recycling processes. For instance, while highly refractory zircons can survive multiple orogenic and sedimentary cycles and transport over extremely long distances, less durable apatites are more likely to represent first cycle detritus and will likely record the thermal history of relatively proximal sources.

In addition to detrital studies aimed at provenance discrimination, multi-dating may be also useful for tectonic studies since it permits more complete and more accurate reconstruction of thermal histories for different minerals and thus provides a potentially powerful tool for exploring the link between deep and shallow processes. The combined application of FT and (U–Th)/He may allow a more robust and more detailed reconstruction of thermal histories within the corresponding partial annealing/retention zones. This is usually achieved using thermal modelling packages like HeFTy or QTQt (Ketcham 2005; Gallagher 2012), which offer a wide range of options and parameters to be defined in order to achieve reliable results. Even though the models have proved viable for reconstructing thermal histories in many studies, caution is recommended when attempting to model a combination of only FT and (U–Th)/He data. In particular, in some situations it is challenging and sometimes even impossible to obtain satisfactory and geologically reasonable results even where the model is constrained by both FT data (age and length) and (U–Th)/He data (i.e. age, size, zonation of parent nuclides, diffusion parameters) (e.g. Danišík et al. 2010b, 2012). While application of inverse thermal modelling often produces geologically reasonable best-fit time–temperature envelopes and paths, there are still challenges and improvements to be made. For example, testing the reliability and reproducibility of modelling results obtained by the combined modelling approach on natural

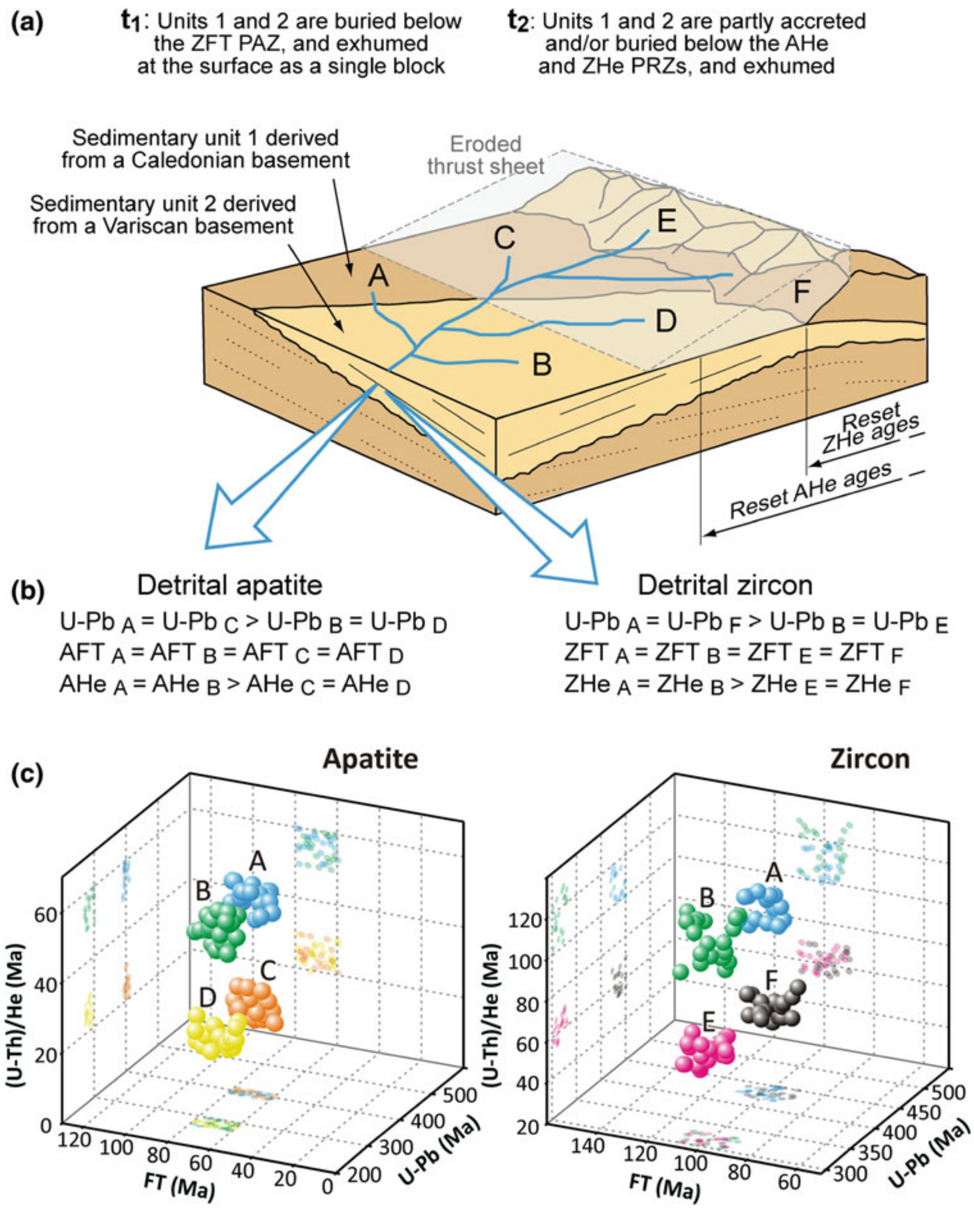

Fig. 5.5 U–Pb, FT and (U–Th)/He data potentially recovered in detritus derived from erosion of a hypothetical geologic landscape shown in (**a**). Detrital apatite and zircon grains eroded from different subareas (A to F) will yield distinct combinations of U–Pb—FT—(U–Th)/He ages (**b**), that can be plotted on a three-dimensional bubble chart (**c**). AFT (ZFT) and AHe (ZHe) indicate FT and (U–Th)/He ages on apatite (and zircon), respectively; PAZ, partial annealing zone; PRZ, partial retention zone

calibration sites or well-characterised samples with a known thermal history (e.g. House et al. 2002).

Finally, triple-dating can help to improve the understanding of FT and (U–Th)/He data and both methods in general. With exception of monazite, the temperature sensitivity of the (U–Th)/He method is generally slightly lower than the sensitivity of the FT method for similar minerals (Fig. 5.1). Therefore, (U–Th)/He ages should ideally be identical or younger than the FT ages obtained on the same mineral. With the exception of correlations between grain size and (U–Th)/He age that are sometimes observed (e.g. Reiners and Farley 2001), (U–Th)/He ages usually do not provide information on the style of cooling through the He partial retention zone. Therefore, in the absence of additional information, it is not evident whether the (U–Th)/He ages are related to a distinct, geologically significant cooling event (and hence can be termed as "cooling" ages), or "apparent" ages without direct geological meaning (e.g. Stockli et al. 2000; Danišík et al. 2015; see Chap. 8 Malusà and Fitzgerald 2018a, b). In addition, single-grain (U–Th)/He ages often show high dispersion that may reflect a number of conditions—a protracted cooling through the He partial retention zone (Fitzgerald et al. 2006), inaccurate alpha ejection correction (Farley et al. 1996; Hourigan et al. 2005), radiation damage affecting He retentivity and closure temperature (Hurley 1952; Flowers et al. 2009; Guenthner et al. 2013; Danišík et al. 2017) or imperfection of dated crystals (e.g. undetected inclusions causing older than expected ages; Farley 2002; Ehlers and Farley 2003; Danišík et al. 2017). Some of these conditions reflect geologically meaningful processes while others result in data that should be evaluated carefully and perhaps disregarded. In this case, provision of U–Pb and FT ages on (U–Th)/He dated crystals provide further constraints on the cooling style and the origin of dispersion.

One of the challenges for modern low-temperature thermochronology remains the issue of "inverted" FT and (U–Th)/He ages (i.e. FT age < (U–Th)/He age) that have been reported from old, slowly cooled terrains (e.g. Hendriks 2003; Lorencak 2003; Belton et al. 2004; Hendriks and Redfield 2005; Green et al. 2006; Danišík et al. 2008; Ksienzyk et al. 2014). This "inverted" relationship, seemingly contradicting the closure temperature concept (Dodson 1973), called into question fundamental concepts of FT annealing and He diffusion, and the reliability of the methods, and prompted renewed interest in methodological research attempting to explain this discrepancy. Due to these apparently younger apatite FT ages versus (U–Th)/He ages, as well as the often large variation of (U–Th)/He single-grain ages from the same sample, enormous progress in the understanding of (U–Th)/He systematics has been achieved. Such work has revealed the importance of phenomena such as radiation damage (Reiners 2005; Shuster et al. 2006; Shuster and Farley 2009; Flowers 2009; Flowers et al. 2007, 2009; Guenthner et al. 2013; Danišík et al. 2017), zonation of parent nuclides (e.g. Meesters and Dunai 2002a, b; Hourigan et al. 2005; Fitzgerald et al. 2006; Danišík et al. 2017) or chemical composition (Djimbi et al. 2015). At the same time, there have also been considerable advances in FT thermochronology, both in methodology and modelling (e.g. Donelick et al. 2005; Enkelmann et al. 2005; Ketcham 2005; Ketcham et al. 2007a, b, 2009; Zattin et al. 2008; Jonckheere and Ratschbacher 2015, and Chap. 4; Gleadow et al. 2018). As a result, many of the (U–Th)/He data sets previously considered as discrepant could be explained. For example, it was shown that the accumulation of radiation damage can increase the closure temperature of the (U–Th)/He system to levels higher than the closure temperature of the FT system (e.g. Shuster et al. 2006; Guenthner et al. 2013; Gautheron and Tassan-Got 2010; Ketcham et al. 2013). Additionally, it was shown that, due to the faster response of the FT system to temperature change, the inverse FT—(U–Th)/He relationship could be a diagnostic indication of short duration heating events such as shear heating along faults (e.g. Tagami 2005) or wildfires (Reiners 2004) (see Chap. 8; Malusà and Fitzgerald 2018a, b). However, there are still situations where FT—(U–Th)/He age relationships are still a matter of ongoing discussion (see Chap 3, Ketcham 2018, Chap. 21, Kohn and Gleadow 2018, and also Hendriks and Redfield 2005, 2006; Söderlund et al. 2005; Green and Duddy 2006; Green et al. 2006; Shuster et al. 2006; Hansen and Reiners 2006; Flowers and Kelley 2011; Flowers and Farley 2012, 2013; Lee et al. 2013; Karlstrom et al. 2013; Fox and Shuster 2014; Flowers et al. 2015, 2016; Gallagher 2016; Danišík et al. 2017). Several examples exist where the "inverted" FT and (U–Th)/He ages have not been satisfactorily explained, and it is not always straightforward whether the discrepancy arises from insufficient understanding of FT annealing, He diffusion or insufficient data quality (e.g. Hendriks and Redfield 2005, 2006; Green and Duddy 2006; Green et al. 2006; Kohn et al. 2009; Danišík et al. 2017). It should be noted that these studies reporting "inverted" FT and (U–Th)/He ages employed a single method or two methods applied to different grains, and this may introduce some bias into the results. In other cases, it was apparent that where (U–Th)/He ages were calculated as "mean ages" they were older than central apatite FT ages, rather than the totality of single-grain (U–Th)/He age data being evaluated in context of various factors such as grain size [eU] and a prolonged cooling history. Application of the in situ triple-dating approach has shown that apparent discrepancies between FT and (U–Th)/He ages do arise simply from such statistical misconceptions in conventional data treatment, where mean or single-grain (U–Th)/He ages were compared with the central apatite FT age, and not with the range of single-grain FT ages (e.g. Danišík et al. 2010a).

In addition to enhancing data interpretation, the routines used in FT dating can be applied to improve the quality of (U–Th)/He dating results. In some FT laboratories, it is common practice to evaluate the suitability of crystals for (U–Th)/He and U–Pb dating based on the sample quality as seen in the FT mounts at high-resolution (~1250×). The high-resolution FT images provide information on crystal size, morphology, appearance and composition of inclusions, degree of radiation damage (e.g. Garver and Kamp 2002) and, perhaps most importantly, the distribution of U (Jolivet et al. 2003; Meesters and Dunai 2002a; Fitzgerald et al. 2006; Danišík et al. 2010a), which is critical for robust alpha ejection corrections (Farley et al. 1996; Hourigan et al. 2005). Although for apatite, U zonation is usually better revealed in induced tracks on mica external detectors, rather than spontaneous fission tracks in apatite grains. Instead of a random selection of grains for further geochronological analysis, such information can be utilised for a targeted grain selection strategy, aiming to represent all sub-populations present in the sample, which should lead to more realistic representation despite fewer grains being dated. Finally, long-term experience has shown that the quality of (U–Th)/He data obtained on samples previously dated by FT methods is better than the quality of data obtained on crystals handpicked and examined under a binocular or a low-magnification petrographic microscope.

Another practical application of FT imaging that simplifies a double-dating approach was reported by Evans et al. (2013). These authors applied SHRIMP U–Pb and conventional (U–Th)/He double-dating to zircons from a diamondiferous lamproite pipe and to detrital zircons from surrounding country rocks, in an attempt to test whether the lamproitic zircon crystals could be identified in detrital population based on their distinctive U–Pb/(U–Th)/He age pattern. The successful outcome of this experiment proved that the double-dating approach is a viable diamond exploration tool, the only practical limitation being the time and labour required to double-date a sufficient number of detrital grains. However, these authors showed that the analytical time of this approach could be dramatically reduced by employing chemical etching of fission tracks in zircon as a pre-screening procedure. Based on their different etching characteristics, this process distinguished lamproitic zircon from the majority of country rock zircons, making the procedure more time- and cost-efficient.

5.6 Concluding Remarks and Future Perspective

This chapter has described the principles, methodologies, applications and advantages of an in situ multi-dating approach in single crystals where the FT method is applied in combination with the U–Pb and/or (U–Th)/He methods. Notably for detrital samples, the first major advantage of multi-dating within a single crystal, as compared to applying a single method, is that data from multiple techniques provides more constraints on the thermal history. The high-temperature U–Pb geochronometer typically records processes at higher temperatures and at greater lithospheric depths, whereas lower-temperature FT and (U–Th)/He thermochronometers are sensitive to thermal changes in upper crust. The second major advantage is that the default provision of three ages from independent radiometric systems on single grains permits a direct internal consistency check of the results, which allows the researcher to identify analytical outliers and thus significantly improve the quality of data for geological interpretation.

Multi-dating has already proven to be a feasible and extremely powerful tool in detrital dating studies, as when detrital crystal geochronological data is obtained by single-dating methods, there may be some inherent ambiguities associated with a lack of geological context. Multi-dating may overcome this limitation by providing critical information about provenance, exhumation, deposition and post-depositional thermal history of source rocks. Finally, the triple-dating approach can be beneficial in solving apparent “inverted” FT and (U–Th)/He age relation issues and therefore offers a useful tool to address the occasional inconsistencies between FT and (U–Th)/He data, which can help to improve the understanding of FT and (U–Th)/He methods in general.

Several analytical procedures can used to obtain combined U–Pb, FT and (U–Th)/He ages on single grains. The most commonly applied approach employs EDM FT dating, U–Pb dating by LA-(MC)-ICPMS and conventional (U–Th)/He dating. This procedure is more efficient when employing FT dating using the LA-ICPMS instead of the EDM FT method where samples are irradiated in a nuclear reactor. An even more efficient approach with higher sample throughput combines FT dating by LA-ICPMS and in situ (U–Th)/He dating, whereby the U–Pb age is obtained as a by-product of LA-ICPMS analysis. Development of this promising concept is currently underway and initial results on the Durango apatite standard are encouraging, which suggests that this approach will be feasible in the future.

Future directions for the triple-dating approach should include the development and optimisation of methodologies and analytical instruments allowing rapid production of high-quality data. Thus far, the triple-dating approach has been successfully applied to zircon and apatite; however, multi-dating methodologies for other minerals (e.g. titanite, monazite, allanite) are yet to be developed and tested. The expected increase of triple-dating studies for detrital sample suites may call for development of new statistical approaches to data deconvolution and identification of principal components in multidimensional space. Last but not least, the reliability of triple-dating data sets and the capability to recover desired age information from detrital grains should be rigorously tested on synthetic samples or well-characterised natural test sites, before triple-dating is applied more broadly.

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