Fission Track Dating of Phosphate Minerals and the Thermochronology of Apatite

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INTRODUCTION

Several phosphate minerals have been investigated for their usefulness in geochronology and thermochronology by the fission track method. Of these, apatite $[Ca_5(PO_4)_3(F,Cl,OH)]$, has proved pre-eminently suitable for this purpose for reasons discussed below. Apatite was one of the first minerals, amongst many others, to be investigated for fission track dating by Fleischer and Price (1964) and has subsequently become by far the most important of all the minerals used for dating by this method. The usefulness of apatites for fission track dating arises from their near-universal tendency to concentrate uranium within their structure at the time of crystallization and their widespread occurrence in all of the major rock groups.

Price and Walker (1963) first recognized the possibility that the accumulation of radiation damage tracks in natural minerals from the spontaneous nuclear fission of ²³⁸U within their lattices could be used for geological dating. They also demonstrated that a simple chemical etching procedure served to enlarge these fission tracks to optical dimensions so that they could be observed and measured under an ordinary optical microscope (Price and Walker 1962b, 1963). This simple procedure quickly opened the way for a wide range of nuclear particle track studies in natural minerals and glasses (e.g., Fleischer et al 1975), the most important of which was fission tracks provided an explanation for various 'anomalous' etch pits in apatite which had puzzled crystal-lographers over many years. The essence of a fission track age determination involves measuring the number of tracks that have accumulated over the lifetime of the mineral along with an estimate of the amount of uranium that is present. Knowing the rate of spontaneous fission decay, a geological age can be calculated.

Detailed studies on apatite for routine geological dating applications began with the pioneering work of Naeser (1967) and Wagner (1968, 1969). These studies established basic procedures that enabled the rapid and widespread adoption of apatite for a variety of fission track dating applications. It was realized very early that fission tracks in minerals displayed only limited stability under exposure to elevated temperatures and that apatite was one of the most sensitive minerals to thermal annealing of fission tracks (Fleischer and Price 1964). Later studies have greatly refined our understanding of track annealing behavior in apatite providing a basis for many of the applications of the method which are discussed below. A comprehensive overview of the general field of fission track dating and its applications has been provided by Wagner and Van den Haute (1992), and a very useful review of methods and interpretive strategies by Gallagher et al. (1998). Here we will focus specifically on the fundamental mineralogical aspects fission of track dating, specifically as applied to apatite and other phosphate minerals, and emphasize the more recent applications and future directions for this field.

FISSION TRACK DATING OF PHOSPHATE MINERALS

Relatively little work has been carried out on fission track dating of phosphate minerals other than apatite, and merrillite, β -Ca₃(PO₄)₂, is the only other example to have received any significant attention. Two early studies examined the potential of monazite, (Ce,La,Y,Th)PO₄, and pyromorphite Pb₅(PO₄)₃Cl, but these minerals have not been studied further. Pyromorphite was shown by Haack (1973) to contain numerous spontaneous fission tracks, but these were very unevenly distributed, making its use in dating very difficult.

Monazite

Monazite is known to contain high concentrations of uranium, so it is perhaps surprising that it has not been investigated further. The only reported fission track dating study of monazite was carried out by Shukoljukov and Komarov (1970) who reported ages from two specimens from Kazakhstan which turned out to be much younger than that expected for the host rock. This observation was attributed to a very low thermal stability of fission tracks in this mineral and consequent fading of fission tracks at relatively low temperatures. Such an observation of reduced fission track age is actually quite typical of the pattern frequently observed in other minerals, particularly apatite, although this was not well understood at the time this study on monazite was carried out.

The apparently low thermal stability of fission tracks in monazite would clearly repay further investigation, as the common occurrence of monazite would otherwise make it an attractive target for fission track analysis. Supporting this view is the general observation that monazite does not become metamict (i.e., have its lattice disordered by the effects of accumulated radiation damage), which is again similar to apatite. This indicates that radiation damage does not accumulate substantially in this mineral, implying that it has a low thermal stability. Monazite fission track dating may therefore have potential as a new low temperature thermochronometer.

Analytical considerations, however, are likely to be the major impediment to monazite dating by conventional fission track methods. These problems arise from the generally very high concentrations of heavy elements in this mineral, and include possible interference from ²³²Th fission due to the very high concentrations of Th present, and the potential for incipient metamictization of the crystal lattice. High concentrations of rare earth elements, typical of this species, could also cause problems of neutron self-absorption by these elements during neutron irradiation (Wagner and Van den Haute1992), leading to an underestimate of the amount of uranium. However if alternative methods were applied to measure U and Th abundances directly then there would seem to be no reason that routine fission track dating could not be applied to this mineral. Such measurements could be achieved by electron microprobe (Montel et al. 1996, Williams et al. 1999) or by laser-ablation inductively coupled plasma mass spectrometry (Cox et al. 2000), making this approach a very fruitful avenue for future research.

Merrillite ("whitlockite")

Merrillite, an anhydrous calcium phosphate mineral often coexisting with apatite in lunar and meteorite samples, has been used for a number of fission track dating studies of extraterrestrial materials. Following a paper by Fuchs (1962) this mineral was most commonly identified in meteorites as whitlockite, but Dowty (1977) has shown that it exhibits significant differences to terrestrial whitlockite, $(Ca,Mg)_3(PO_4)_2$, and should be distinguished from it. As a result, earlier publications use 'whitlockite' while later ones apply the name 'merrillite', for the same mineral. Merrillite is now the appropriate species name for the high-temperature phosphate mineral found predominantly in meteorites.

Fission track dating of merrillite has some unusual characteristics compared to the dating of terrestrial apatites due to the great age of these extraterrestrial samples. The fission track densities are extremely high, requiring modified track etching and counting procedures (e.g., Crozaz and Tasker 1981). Typically the tracks are very lightly etched compared to what would be used for normal observation in terrestrial minerals. As a result the tracks are very small and must be counted using scanning electron microscopy, either on the etched material itself or on a plastic replica of that surface, rather than by optical microscopy. An example of an etched merrillite grain from Apollo 12 lunar rock 12040 is shown in Figure 1 (Burnett et al. 1971).



Figure 1. Scanning electron microscope image of the etched surface of a merrillite grain in lunar rock 12040. The sample has been very lightly etched in 0.12% HNO₃ for 20 s revealing an extremely high density of spontaneous fission track etch pits (numerous small dark dots). The grain has a considerable excess of ²⁴⁴Pu tracks as well as ²³⁸U tracks giving it an apparent fission track 'age' of 6.4 Ga. [Used by permission of the publisher, from Burnett et al. (1971) *Geochimica et Cosmochimica Acta*, Supplement 2, p. 1507.]

Two other factors unique to these extraterrestrial samples must also be accounted for. The first is that tracks from heavy cosmic ray particles (mainly Fe-group nuclei) and other nuclear interactions with cosmic rays may also be present, in addition to fission tracks. The simplest method for correcting for the cosmic ray background is to measure the track density in adjacent silicate mineral grains, such as feldspar and pyroxene, which do not contain uranium and are therefore free of fission tracks. The second factor is that the samples are so old as to contain tracks from the spontaneous fission of now-extinct transuranic elements, particularly ²⁴⁴Pu with a half-life of 82 Myr. Such Pu tracks will only be present in samples older than about 3.9 Gyr (Crozaz and Tasker 1981).

The occurrence of extinct ²⁴⁴Pu tracks first became apparent when a large excess of fission tracks over that which would be expected from ²³⁸U spontaneous fission alone was discovered (e.g., Burnett et al. 1971). If calculated as a fission track age in the usual

way, assuming all tracks were produced by ²³⁸U spontaneous fission, these data led to apparent ages which were considerably greater than the age of the Solar System. Significantly an excess of tracks is not always observed in chlorapatites from similar lunar materials. In the case of some Apollo 12 lunar samples, apatites gave much younger fission track ages of only about 1,300 Myr (Burnett et al. 1971). It may be that the apatite ages represent a genuinely much younger age for the rock or the time of heating by a later thermal event. Another possible explanation would be that tracks in merrillite may be stable to higher temperatures than in apatite and so have survived later heating sufficient to substantially reduce the number of fission tracks in apatite. Mold et al (1984) have provided some experimental evidence that tracks in merrillite may indeed be stable to somewhat higher temperatures than in co-existing chlorapatites, but the difference is probably not significant over geological timescales.

A different explanation was provided by Pellas and Storzer (1975) who showed that uranium is more enriched in apatite than in merrillite, at least in meteorites, whereas plutonium is preferentially concentrated in merrillite. This would explain why a much greater ²⁴⁴Pu track excess is typically observed in merrillites without the need for complex thermal histories. Burnett et al. (1971) found that in some Apollo 12 lunar samples, apatites and merrillites had similar uranium concentrations and generally smaller plutonium track excesses than are typically observed in meteorite phosphates. This difference may reflect the generally younger ages of the lunar rocks examined compared to most meteoritic materials. These observations have important implications for the early history of the lunar surface implying the formation and survival without later heating of crustal materials from the first few hundred Myr of its existence as a planetary body. Similarly, the retention of plutonium tracks and their relatively low thermal stability in merrillite have provided useful information about the cooling rates of meteorites in the early history of the solar system (e.g., Pellas et al. 1983, Storzer and Pellas 1977).

Apatite

Apatite initially accounted for a relatively minor fraction of the number of fission track age determinations, representing only about 20% of all measurements by the early 1970s, at which time natural glasses and micas were the most widely used materials. This fraction grew to around 40% by the mid 1980s, over 50% by 1990 and is probably in excess of 70% today. This increasing proportion is even more significant in that the total number of fission track analyses published has increased markedly throughout this period. The striking pre-eminence of apatite amongst the wide variety of minerals that have been utilised for fission track dating is because of the degree to which it consistently meets all of the following criteria. To be widely useful for routine fission track dating a mineral should be:

- well crystallized
- consistently enriched in uranium
- significantly larger in grainsize ($>\sim 60\mu m$) than fission track dimensions
- uniform in its uranium distribution
- highly transparent and free of obscuring inclusions, and
- of common occurrence in all major rock groups.

Crystalline apatite meets all of these criteria extremely well, particularly the last which is a vitally important, but often under-appreciated, factor for any mineral to achieve widespread application. Many other minerals have been the subject of overly optimistic reports on their suitability for fission track dating but fail meet one or more of these requirements and so are limited in their application. For example, cryptocrystalline apatite has generally proved unsuitable because the grain size is too small and etching along grain boundaries obscures any tracks present. Any mineral that is not sufficiently common will always be severely limited in its application to real geological dating problems. Apatite on the other hand is a ubiquitous primary accessory phase in a wide variety of igneous and metamorphic rocks and is also a common detrital mineral in most clastic sedimentary rocks. For example, experience has shown that useful amounts and grain-sizes of apatite can be extracted from approximately 80% of sandstones (Dumitru et al. 1991).

Several additional characteristics of apatite have contributed to its dominance in fission track dating. These include its simplicity of handling in terms of mineral separation, mounting, polishing and etching procedures; its reproducible etching characteristics; and its well-characterized and reproducible response to elevated temperatures. It also has excellent optical characteristics and is mostly free of interfering inclusions and dislocation etch pits providing excellent conditions for observing fission tracks. Importantly apatite is also resistant to the effects of chemical weathering (Gleadow and Lovering 1974) which makes it a remarkably persistent detrital phase in sandstones, except where these have been affected by low pH groundwaters (Morton 1986, Weissbrod et al. 1987). Tracks in apatite are also resistant to the effects of pressure and brittle deformation (Fleischer et al. 1965a). Finally, apatite does not accumulate radiation damage from alpha decay and so does not become metamict. Incipient metamictization progressively modifies the properties of the next two most commonly dated minerals by the fission track method, zircon and titanite, adding complexity to their processing and limiting their applicability, especially in older rocks.

For all these reasons apatite can be considered an ideal mineral for fission track dating. It can be extracted in useful quantities from the great majority of crystalline and clastic rocks, and almost always contains suitable quantities of uranium (typically 1-50 μ g/g). This means that it can be applied to a wide variety of geological studies, especially those requiring a significant regional coverage of sampling, and has thus become the standard mineral for the great majority of fission track dating studies. Apatite will be the only mineral considered further in this article.

FISSION TRACK DATING OF APATITE

The formation of fission tracks

In addition to the extinct isotope ²⁴⁴Pu discussed above, the very heavy isotopes ²³²Th, ²³⁵U and ²³⁸U all undergo spontaneous nuclear fission and remain in existence. In practice, only ²³⁸U is a significant source of natural fission tracks in terrestrial minerals as it has a fission half-life of 8.2×10^{15} years (see Wagner and Van den haute 1992). While extraordinarily long, this is still 4 to 6 orders of magnitude shorter than the spontaneous fission half-lives of 232 Th and 235 U. In terrestrial apatites, therefore, it can safely be assumed that solely ²³⁸U has produced all the fission tracks observed. These three heavy isotopes also decay through chains of alpha (⁴He) and beta emissions to produce three different isotopes of lead. The alpha decay half-life is very much shorter for each of these isotopes than for fission so that for ²³⁸U there are more than 10⁶ alpha decays for every fission event. However the alpha decay events do not themselves cause sufficient defects to produce etchable tracks and appear to produce no other permanent accumulation of lattice damage in apatite. It is worth emphasizing here that each spontaneous fission track represents a single ²³⁸U nuclear fission event. It is this extreme sensitivity for the detection of individual nuclear events that makes the fission track dating method practical, despite the extraordinarily long half-life for the fission decay, which is around six order of magnitude longer than for the commonly used isotopic dating methods.

The mechanism of track formation is a combination of two simultaneous and very transient processes. Upon disintegration of the parent uranium nucleus, the fission fragments, with excess kinetic energy, are propelled from the reaction site in opposite directions. Each high-energy fragment interacts with the host lattice, fundamentally changing the properties of the atomic structure. The nature of this interaction changes as the particle loses energy and slows down Paretzke (1986). Total fragment energy imparted to the solid is the sum of both electronic and nuclear interactions by the fragments, in addition to some emitted radiation. Electronic stopping occurs when the fast moving ion undergoes inelastic collisions with lattice electrons. The number of these collisions is large and with each interaction the particle surrenders energy by stripping electrons from target atoms, by having its own electrons stripped away, and by raising the excitation levels of lattice electrons. As the projectile slows, nuclear stopping becomes more important. This regime is dominated by elastic collisions between two free particles (projectile ion and target atom) at low velocities, hence the "billiard ball" analogy (Kelly 1966). The ionization level of the projectile ion diminishes with continued loss of energy and by the time nuclear stopping becomes dominant, the ion is in a neutral state.

As Wagner and Van den haute (1992) pointed out, there is general agreement on the nature of the processes affecting the fission fragment as it slows and loses energy in a solid. What is less clear and has long been the subject of some debate is the nature of the processes suffered by atoms of the crystalline solid. Some authors (Vineyard 1976, Chadderton 1988, Spohr 1990) have supported a model widely accepted in ion implantation research: the "thermal spike". Here the term "spike" evokes the sense of a rapid, intense event and the term has found favor with many descriptions of the track formation process (Vineyard 1976, Fleischer et al. 1975, Chadderton 1988, Spohr 1990). The thermal spike concept treats the rapid deposition of energy into the lattice as a near-instantaneous heating event with a time span on the order of 10^{-12} seconds. According to the model, intense heat and multiple collision cascades generate a plasma in the track core (Wein 1989) followed by transport of the energy to the surrounding lattice by heat conduction processes (Chadderton 1988). Lattice defects created during the thermal activation process are quenched within the solid as the thermal perturbation rapidly decays (Spohr 1990) and the track core is left in a massively disordered state (Fleischer et al. 1975).

An alternative track formation model has long been accepted amongst fission track researchers (Wagner and Van den haute 1992). The "Coulomb explosion" model or "ion explosion spike", (Young 1958, Fleischer et al. 1965a,b; 1975), as its name suggests, is largely electrostatic in nature and explains the primary observation that nuclear tracks are only observed in dielectric solids. The massive positive charge on a rapidly moving fission fragment strips and excites lattice electrons along its path through the lattice (in this case apatite) leaving a core of positively-ionized lattice atoms. The resulting clusters of positive ions then explosively recoil away from each other as a result of coulomb repulsion as illustrated in Figure 2. Secondary cascades of atoms result from the barrage of ionized atoms that have been discharged from the track core. This is consistent with the results of small angle neutron experiments in other minerals, in which a significant density deficit within the track core is observed (Albrecht et al. 1982, 1985). The displacement cascades generate profuse interstitials and vacancies, which in turn cause distortions and elastic strain gradients within the lattice structure. This, it is argued, is responsible for the amorphous nature of lattice damage seen in latent/unetched tracks that have been examined by electron microscopy in apatite (Paul and Fitzgerald 1992).



Figure 2. Three main stages of the ion explosion spike according to Fleischer et al. (1975). (a) The highly charged fission fragment ionizes lattice atoms along its trajectory. (b) Electrostatic repulsion causes displacement of lattice atoms along fragment path. (c) The matrix is strained elastically in proximity to defects and defect clusters. Some relaxation occurs in the lattice.

Track etching and observation

Apatites are usually prepared for fission track analysis by mineral separation from the host rock using conventional heavy liquid and magnetic techniques on a 60-200 µm size fraction of crushed rock. A sample of apatite crystals, usually several hundred grains, are then mounted in an epoxy film on a glass slide and mechanically ground and polished to reveal an internal surface cut through the interior of the grains. The tracks are then simply revealed by etching the polished mount in dilute HNO₃ (most commonly 5N) at room temperature for 20 seconds, although minor variations of etchant concentration and time are used in different laboratories. The form of the track etch pits varies according to the orientation of the polished surface relative to various crystallographic directions (Wagner 1968, Wagner and Van den Haute1992).

A preference was shown by some early workers for observation of fission tracks on basal surfaces of apatite under oil immersion. Tracks on these surfaces have an unusual and distinctive form, consisting of a broad hexagonal pit with a narrow track channel continuing into the crystal from the apex (Wagner 1968, 1969). Subsequently most workers have adopted observation using dry microscope objectives $(100\times)$ and using surfaces roughly parallel to prism faces (Gleadow et al. 1986) at a total magnification of 1000× or greater. Such observation conditions have several advantages, and are particularly favored for the external detector method of analysis that will be discussed below. Although oil immersion objectives are capable of higher optical index resolution, the refractive of immersion oil (n = 1.54) is very close to

that of the apatites themselves, drastically reducing the optical contrast of the tracks relative to the host material. Dry objectives provide a substantially higher contrast in the image of fission tracks in apatite making them much easier to observe (Gleadow et al. 1986). In addition, dry objectives are much more convenient, particularly with motor drive microscope stage systems.

The typical prismatic habit of most apatite crystals means that they tend to align on prism faces during mounting in the epoxy mounting medium, so that after subsequent polishing many of the new surfaces exposed are roughly parallel to the *c*-axis. This means that a substantial fraction of the grains have surfaces which are in a consistent crystallographic orientation and reveal tracks as elongated narrow channels. Etching is usually continued until the tracks are approximately 1-2 µm in diameter as progressive etching experiments have shown that the number of tracks revealed is stable when counting at these dimensions (Gleadow and Lovering 1977). The appearance of well etched fission tracks on a polished surface of an apatite crystal is shown in Figure 3. Further etching reveals that the tracks have the form of faceted etch pits related to the underlying symmetry of the crystal as shown diagrammatically in Figure 4. Tracks on surfaces parallel to the crystallographic *c*-axis are needle-like when first revealed, and take on the form of faceted knife-blades with continued etching (Wagner 1969), contrasting with the broad hexagonal pits observed on basal surfaces (Fig. 4). These forms reflect the anisotropy of etching which follows different crystallographic directions. Even though the tracks are manifestly anisotropic in their etching behavior they appear to have a uniform distribution with angle to the *c*-axis so that tracks at all orientations are revealed satisfactorily for identification and counting.

Some spurious, non-track features, such as dislocations, may also be revealed by etching in apatite. Although these may be superficially similar to fission tracks in appearance, and very occasionally may may cause serious interference to track counting



Figure 3. Etched fission tracks in an apatite grain from the Grassy Granodiorite of King Island, southeastern Australia. The tracks show their characteristic appearance as randomly oriented, straight-line etch channels up to a maximum length of around 16μ m. The tracks appear as dark, high-contrast features in this image observed under a high magnification dry objective. Faint continuous lines across the surface are polishing scratches.

Figure 4. Schematic diagram showing the form of strongly-etched fission tracks etched on different crystal surfaces in apatite. The anisotropic etching which is characteristic of apatite produces knife-blade shapes for tracks on the preferred prismatic faces. Such features are only obvious after much longer etching times than usually applied for track counting.





Figure 5. Dense swarms of dislocation etch pits in two apatite grains from the Early Cretaceous sandstones of the Otway Basin of southeastern Australia. Grains containing such high dislocation densities are most often observed in volcanic apatites and are best completely avoided for fission track counting. Typical dislocation features such as sub-parallel arrays, branching and extremely long etch channels can be seen. Fortunately, even in such samples, many grains are free of such features enabling reliable track counting.

(Fig. 5, above), tracks can be reliably discriminated from spurious etch features in that tracks are straight-line defects which are of limited length and randomly oriented. Dislocation etch pits, on the other hand, tend to be wavy or curved, are often of much greater length than tracks, frequently show branching, and often occur in swarms of parallel or sub-parallel features (Fig. 5). Such grains are best avoided altogether. Dense dislocation swarms are found most commonly in volcanic apatites but are extremely rare in apatites of plutonic origin, an observation that has not yet been fully explained. The distinction may relate to the different thermal histories of these two groups of apatite. Fortunately, however, the great majority of apatite grains, are almost entirely free of dislocation etch pits, even in the rock that produced the extreme examples in Figure 5. Therefore the selection of grains in which fission tracks can be identified is relatively straightforward and reliable in most apatite separates.

Fission track dating methods

The basic principles and practical methods of fission track age determination have been described elsewhere (e.g., Fleischer at al. 1975, Wagner and Van den Haute1992, Gallagher et al. 1998) and will be summarized only briefly here. Once fission tracks have been revealed by etching, the main parameter to be measured, which is representative of geological age, is the track density or the number of tracks per unit area on the etched surface. This is measured by counting the number of track intersections with the surface using a calibrated grid in the microscope eyepiece. For a given uranium concentration, the spontaneous fission track density will steadily increase through time, provided the tracks remain stable and are therefore quantitatively retained.

Determination of a fission track age requires several further experimental steps to measure the uranium concentration. The uranium concentration is not measured directly, but a second set of fission tracks is created artificially in the sample by a thermal neutron irradiation. This irradiation induces fission in a tiny fraction of the ²³⁵U atoms, which are present in a constant ratio to ²³⁸U in natural uranium. Knowing the total neutron fluence received during irradiation, the number of induced tracks provides a measure of the uranium concentration of the grain. Because the induced tracks are derived from a different isotope of uranium than the spontaneous tracks an important consideration in fission track dating is the assumption that the isotopic ratio of the two major isotopes of uranium, ²³⁵U and ²³⁸U, is constant in nature. With the notable exception of the unique "natural" nuclear reactors of Oklo in Gabon (Bros et al. 1998), where this isotopic ratio is disturbed, this is a very safe assumption. Numerous measurements have shown that ²³⁵U and ²³⁸U are always present in their natural abundances of 0.73% and 99.27%, respectively.

The fission track age, t, is then calculated from the ratio of spontaneous (ρ_s) to induced (ρ_i) track densities according to the standard fission track age equation (Fleischer and Price 1964, Naeser 1967):

$$t = \frac{1}{\lambda_D} \ln \left(1 + \frac{\lambda_D \phi \sigma I}{\lambda_f} \frac{\rho_s}{\rho_i} \right)$$
(1)

where λ_D is the total decay constant for ²³⁸U from all decay modes (effectively just the α decay constant), λ_f is the fission decay constant, ϕ is the total neutron fluence received, σ is the thermal neutron cross section for ²³⁵U, and I is the ²³⁵U/²³⁸U isotopic ratio for natural uranium (effectively a constant in nature). The neutron fluence is most conveniently measured by determining the induced track density, ρ_d , produced in a calibrated uranium-bearing glass irradiated along with the dating samples. The fluence is related to ρ_d , usually measured in an mica external track detector held adjacent to the standard glass, by the following:

$$\phi = B\rho_d \tag{2}$$

where B is a constant of proportionality. Substituting (2) into Equation (1) gives:

$$t = \frac{1}{\lambda_D} \ln \left(1 + \lambda_D \zeta \frac{\rho_s}{\rho_i} \rho_d \right)$$
(3)

where

$$\zeta = \sigma I B / \lambda_f \tag{4}$$

The aggregate constant ζ (zeta) is determined empirically by measurements on age standard materials following the initial suggestion of Fleischer and Hart (1972), subsequently elaborated by Hurford and Green (1982, 1983) and Green (1985). In principle it is possible to determine the component constants individually and calculate the ages absolutely (Wagner and Van den Haute1992) although various experimental difficulties with this approach have led to the emergence of the zeta calibration. The empirical zeta approach has been recommended by the IUGS Subcommission on Geochronology (Hurford 1990) and since adopted almost universally. The various issues involved in thermal neutron irradiation were discussed in detail by Green and Hurford (1984).

Experimental procedures

Measurement of a fission track age following Equation (3) requires the determination of three different track densities, ρ_s , ρ_i and ρ_d . The various different experimental strategies involved have been elaborated by Naeser (1979a), Gleadow (1981), Hurford and Green (1982) and Wagner and Van den Haute(1992) and are summarized in Figure 6. Of the five main alternatives, only the Population (PM) and



Figure 6. Schematic representation of different fission track dating procedures (after Hurford and Green 1982). Of these, only population and external detector methods have gained wide currency.

External Detector (EDM) methods have been extensively used for apatite and the EDM has now become the standard procedure in most laboratories. Galbraith (1984) describes the statistical treatment of analytical data derived by both these methods, and interlaboratory comparisons have mostly demonstrated excellent reproducibility using both procedures (Miller et al. 1985, 1990).

The Population Method measures both ρ_s and ρ_i on internal surfaces within the apatite grains themselves, but on two separate aliquots, assuming that the uranium concentrations of the two aliquots are statistically equivalent. Whereas the PM was initially the preferred method for dating apatites (e.g., Naeser 1967, Wagner 1968) the EDM is now usually preferred because it provides age information on a grain-by-grain basis. The variability between single grain ages has turned out to be important (Galbraith and Green 1990, Galbraith and Laslett 1993), as expected in the case of dating detrital apatites from sedimentary rocks, where a spread of single grain ages could be anticipated, but surprisingly, also in application to igneous rocks (e.g., O'Sullivan and

The steps involved in the External Detector Method are illustrated in Figure 7. The spontaneous tracks are etched on an internal polished surface on the apatite grains and the induced tracks on a mica external detector attached to the grain surface during neutron irradiation. After irradiation the external detector is etched to reveal an induced fission track image corresponding to grains in the apatite mount. Even though this results in a second set of tracks being produced within the apatite grains themselves, these tracks will not be detected because they are not etched after the irradiation.



Figure 7. The sequence of steps involved in the external detector method of fission track dating. This method is now the dominant procedure used in most fission track dating laboratories for apatite because of its ease of handling, suitability for automation and its provision of single grain age information.

Parrish 1995).

To measure the fission track age by the EDM involves determining ρ_s in a selected apatite grain and finding the mirror image area on the mica external detector where ρ_i is counted over exactly the same area. Because the geometry of track registration is not the same for the internal surface (4π) on which the spontaneous tracks are measured and the external detector surface (2π) used for induced tracks, a geometry factor must be introduced to correct for this difference. The geometry factor is ~0.5, but not exactly because of small differences in etching efficiency on the two surfaces and differences in the range of fission tracks in the two different materials (e.g., Gleadow and Lovering 1977, Green and Durrani 1978, Iwano et al. 1993). An individual age can be calculated for each grain counted using Equation (3). A combined age for the sample may also be calculated, usually using the 'central age' of Galbraith and Laslett (1993), which is a weighted mean of the log normal distribution of single grain ages. Errors on ages are usually calculated using the 'conventional method' described by Green (1981),

The EDM is broadly applicable to most minerals used in fission track dating and also allows for a high degree of automation to be applied. Computer-controlled microscope stage systems are now usually used for the purpose of matching areas of induced tracks on the mica external detector to the corresponding grains in the apatite mount (Gleadow et al. 1982, Smith and Leigh-Jones 1985, Crowley and Young 1988). Such systems provide a greatly enhanced productivity compared to older manual methods of grain location and additionally present an opportunity for systematic organization and management of data collection.

Track length measurements

Implicit in Equation (1) above is an assumption that the track lengths of spontaneous and induced tracks are the same. This is because the 3-D distribution of fission events within the apatite crystal is related to the observed track density on a 2-D surface via the average track length. In practice the lengths of spontaneous and induced tracks are never exactly the same for apatite (Gleadow et al. 1986) due to some shortening of the spontaneous tracks over their lifetime. As a result, some knowledge of the distribution of track lengths is essential in order to interpret properly the apparent fission track ages obtained.

From each fission event, the two fission fragments travel in exactly opposite directions to produce a single linear damage trail with an overall length equal to the combined range of both particles. The *etchable* length of each track is actually somewhat shorter than the combined range as there is a small 'range deficit' of up to several µm at each end of the track where the damage intensity is not sufficient to produce a continuous etchable track (e.g., Fleischer et al. 1975, Iwano et al. 1993). On an internal surface of an apatite crystal tracks at all lengths up to the maximum etchable length are observed, with the shortest tracks produced from a fission event almost one fission fragment range above the surface (e.g., Gleadow and Lovering 1977). Clearly such surface-intersecting tracks are randomly truncated at some arbitrary distance along their lengths, making their use in estimating the full etchable length difficult.

Several track length parameters have been utilized in fission track dating studies since the first use of projected track lengths in apatite by Wagner and Storzer (1972). They were the first to recognize that spontaneous tracks had different length distributions relative to fresh induced tracks in apatite. Dakowski (1978) established a clear understanding of the geometric properties of the various length parameters which was further extended by the work of Laslett et al. (1982). Following the work of Bhandari et al (1974) it has been shown that the greatest information about the true distribution of fission track lengths can be obtained from the measurement of horizontal "confined" tracks (Laslett et al. 1982, Gleadow et al.1986). Confined tracks do not intersect the

polished surface, but are etched wholly within the body of the mineral where the etchant has gained access below the surface along other tracks or fractures. These have been identified as Track-in-Track (TINT) or Track-in-Cleavage (TINCLE) events by Lal et al. (1969). Several examples of such confined fission tracks, are shown in Figure 8 and procedures for measuring their lengths are described in detail by Gleadow et al. (1986).



Figure 8. Etched spontaneous fission tracks on a polished internal surface cut in an apatite crystal, observed at high magnification using a dry microscope objective. Most of the visible tracks are surface intersecting spontaneous tracks which are used for age determination. Arrows point to four individual *confined tracks* which do not intersect the surface but are fully contained within the body of the apatite crystal and etched from fractures which allow passage of etchant from the surface to the tracks. Such confined tracks are used for length measurement and provide the closest approximation to the true distribution of etchable lengths of latent, unetched fission tracks. The center pair of confined tracks also illustrate the effect of anisotropic etching with the track across the grain being much wider than the narrow track which lies closer to the elongated c-axis of the crystal. After Gleadow et al. (1986).

The application of fission track length studies to the interpretation of fission track ages depends on three properties of spontaneous fission tracks.

- 1. All tracks in apatite have a very similar initial length (Gleadow et al. 1986), which is controlled by the energetics of the fission decay and the nature of the track recording material (e.g., apatite).
- 2. Tracks become progressively shorter during exposure to elevated temperatures so that the final length is controlled principally by the maximum temperature that each track has experienced.
- 3. New tracks are continually added to the sample through time so that each one has experienced a different fraction of the total thermal history.

These factors combine to give a final distribution of track lengths which contains a complete record of the temperatures experienced, below about 120°C. Different length distributions result from different styles of thermal history, as illustrated in Figure 9.



Figure 9. Predicted fission track age and length distributions resulting from three different hypothetical cooling histories, all cooling within the same temperature interval between 120° and 20° C. The resulting length distributions are shown as the three histograms on the right. The three numbers on each histogram represent the predicted fission track age (top), the mean track length, and the standard deviation of the length distribution (bottom). Case (a) shows an example of rapid cooling which results in an event age closely approximating the time of rapid cooling, (b) a steady cooling path leading to a broader, skewed distribution and a younger cooling age, and (c) a two-stage cooling history resulting in a mixed age and a bimodal track length distribution. Modified after Gleadow and Brown (2000).

When combined with the apparent fission track age, length distributions can be used to reconstruct the variation of temperature through time.

Observations of track lengths from a wide variety of surface rocks (Gleadow et al. 1986) show that distinctive patterns of track length characterize particular geological environments. Figure 10 illustrates the major length distribution categories identified by Gleadow et al. (1986) in rocks for which the thermal history is known, or can be inferred with reasonable accuracy. The undisturbed volcanic type is, as its name implies, characteristic of volcanic rocks that have remained undisturbed and at relatively low surface temperatures since their formation. A similar pattern will result in any rock which has cooled rapidly and not been re-heated thereafter. This type of distribution is similar to that shown by fresh induced tracks, although the mean length is slightly lower, indicating that some shortening occurs in spontaneous tracks even at ambient surface temperatures.

Apatite grains that have spent a significant period of time within the fission track annealing zone will show various patterns of broader length distribution, such as the undisturbed basement type, representing monotonic cooling from temperatures above about 120°C. More complex, multi-stage thermal histories will produce the even broader 'mixed' distributions. When the peaks in such a distribution are clearly resolved, as in the bimodal case, the distribution is indicative of a two-stage history with an older generation of tracks shortened during a later thermal event, and a new generation of long tracks produced subsequently. Such a bimodal distribution is particularly useful, giving information on the timing as well as the severity of the thermal event.

TRACK STABILITY

The phenomenon of fission track fading, or annealing, was first recognized by Silk and Barnes (1959) who showed that materials that had undergone heating contained shorter tracks than untreated specimens. The fading mechanism was initially interpreted



Figure 10. Representative track length distributions for spontaneous tracks in the various apatite length groups recognized by Gleadow et al. (1986). The top row represents measurements on horizontal confined tracks for which the differences between the different types are more distinctive than for the corresponding projected length distributions (bottom row). 100 confined tracks and 500 projected track lengths were measured in each case. After Gleadow et al. (1986).

by Fleischer et al. (1964) as a pinching out of segments along the track which prevented further access of the track etchant. Naeser (1979a) suggested that the diffusion rate within a heavily annealed track gradually approached that of the undamaged solid, so that it became increasingly difficult to etch out the track. It remains unclear precisely how the structure of the track, at the atomic level, may be altered in response to changes in environmental conditions, such as pressure, ionization, increased temperature, and duration of exposure (Fleischer et al. 1965b), but it is now generally accepted that temperature and time are the primary controls on track stability. Experimental studies by Green et al. (1986) confirmed that track fading was probably a two-part process, in which tracks at first begin to shrink from each end, while continuing to be etchable for the remainder of their length. Eventually track fading enters another phase, one of segmentation (Green et al. 1986), where the latent track cannot be fully etched and is apparently broken by small unetchable gaps (see also Hejl 1995). Transmission Electron Microscope observations of highly annealed tracks (Paul and Fitzgerald 1992).

A fission track has its inception when two fission fragments pass violently through the crystal lattice at high velocities with an initial energy of around 1 MeV/nucleon (Fleischer et al. 1975). The metastable damage zone formed (Fig. 2) immediately begins to heal (Green 1980, Donelick et al. 1990) at a rate largely determined by the temperature of the sample and, to a lesser extent, the duration of the elevated temperature (Laslett et al. 1987). The tracks will continue to shorten until they cool to lower temperatures. The final length of each track therefore represents the integrated result of its passage through time-temperature so that each, in effect, behaves as maximum-recording thermometer.

Progressive shortening of the confined track length is accompanied by a reduction in the measured track density. This is due to the reduced probability of shortened tracks intersecting the polished surface of a grain and thus being exposed to the etchant. As the observed or apparent age of samples is determined on the basis of track density (Naeser and Faul 1969, Wagner and Reimer 1972, Nagpaul et al. 1974) considerable research aimed at determining the reduction of track density during annealing has been reported (Wagner and Storzer 1972, Bertel and Mark 1983, Laslett et al. 1984, Green 1988). The term "apparent" is emphasized since the observed age may be modified by the amount of track annealing that has occurred.

Variations in the observed fission track age with track fading initially seemed to be something of an impediment to mineral dating by this method. However, the systematic and progressive nature of the track length reduction presented an unparalleled opportunity to extract thermal history information in addition to the chronology (Wagner and Storzer 1972). Track length distributions observed in geological samples, were seen to be the net result of both track production and track fading processes over a span of geological time. Indeed, it was clear that there was a wealth of information available if the natural track distributions could be understood.

Annealing over geological time-scales

Typical crustal geothermal gradients are around $20-30^{\circ}$ C/km so that the temperature at 4-5 km depth is in the range 100-120°C, allowing for surface temperatures of around 10-20°C (Pollack et al. 1993). The analysis of samples from deep bore holes (e.g., Naeser and Forbes 1976, Naeser 1981, Gleadow and Duddy 1981, Hammerschmidt et al. 1984), has provided direct evidence of natural thermal annealing of fission tracks in apatite over geological time-scales. Data from hydrocarbon exploration wells drilled within the Otway basin in southeastern Australia (Gleadow et al. 1983, Green et al. 1989a) clearly demonstrate a systematic reduction in the mean confined track length and apparent fission track age with increasing temperature (Fig. 11). At temperatures greater than about 120°C (depths > ~3km) no fission tracks are preserved within apatite and so the apparent fission track age and mean length are effectively zero. Both the apatite fission track age of ~125 Myr at the surface to zero at a depth of ~3.5 km forming a characteristic concave-up profile of apparent apatite age (Fig. 11).



Figure 11. Composite apatite fission track crustal profiles of mean fission track length (\bullet) and apparent apatite fission track age (\bigcirc) plotted against depth for samples from several wells from the central Otway Basin in southeastern Australia. These clearly illustrate the progressive decrease in mean track length and apparent apatite fission track age with depth, and the characteristic concave-up form of both profiles. After Gleadow and Duddy (1981b) and Green et al. (1989a).



Figure 12. Fission track length distributions, single crystal age histograms and single crystal age radial plots (Galbraith 1990) for four individual samples from the central Otway Basin wells (data in Fig. 11). These are representative of successive degrees of thermal annealing and illustrate the progressive change in the shape of the track length distribution and dispersion in apparent single crystal fission track ages with increasing depth. The mean apparent age and the mean track length of the sample decreases with progressive thermal annealing from its original value of approximately 120 Ma. In addition the dispersion of track lengths and single crystal apparent ages increases as the degree of thermal annealing increases. This occurs because fission tracks in the individual apatite crystals anneal at different rates due to the effect of variable chemical composition and annealing anisotropy. After Green et al. (1989a), Brown et al. (1994), and Gallagher et al. (1998).

Four representative track length distributions for different depths in these wells are shown in Figure 12. The observed increase in the standard deviation of the length distribution with decreasing mean track length (higher T) is a consequence of the anisotropy of track shortening (tracks perpendicular to the *c*-crystallographic axis anneal faster than tracks parallel to the *c*-axis) as well as the variation in apatite composition between grains (e.g., Green et al. 1985, Green 1988). The thermal history for Otway

basin samples can be reconstructed from the relatively simple burial history and indicates that this pattern was produced by heating times in the range of 10-100 Myr. Also shown in Figure 12 are a series of histograms of single grain ages with smoothed probability distributions and radial plots (Galbraith 1990) from the same four samples (Dumitru et al. 1991, Gallagher et al. 1998).

Other examples of natural thermal annealing can be seen in the vicinity of shallow level igneous intrusions. Calk and Naeser (1972), for example, demonstrated a systematic reduction in the apparent apatite fission track age of an 80 Myr old granitic pluton with increasing proximity to the contact with a small (\sim 100 m) basaltic intrusion emplaced \sim 10 Myr ago. This pattern of age reduction within the granite was influenced by the thermal effect of the basalt intrusion and is consistent with the pattern of annealing observed in laboratory annealing experiments and deep drill holes.

In order to improve our understanding of the relationships between time, temperature and the observed track parameters in the natural environment, numerous experiments have been conducted over the last twenty-five years. Perhaps the most useful outcome of these annealing experiments has been the development of robust mathematical modeling.

Ever-more sophisticated models continue to be developed, from which geological interpretations of greater precision can be extracted. We will now examine some of the laboratory annealing experiments, as well as some of the current ideas on the processes involved, highlighting some the strengths and the weaknesses of the approach, and describing several problems that have been overcome to make the fission track technique a versatile and powerful tool in tectonic and landscape analysis. This review of some of the key developments in apatite annealing as it pertains to fission track thermochronology includes a selective and by no means exhaustive bibliography.

The process of annealing

When a solid contains defects that exceed the equilibrium concentration, as in the case of radiation or fission induced damage, the defects will react to reduce the free energy of the solid (Kelly 1966). These reactions include diffusion of defects to sinks, immobilization at traps, annihilation, and complexing and clustering with other defects such as vacancies, interstitials or impurities that may occur within the sample (Fig. 13), processes that are diffusion-limited rather than reaction-limited. The speeds of the reactions are controlled by the rate at which a defect species can move through the solid crystal lattice (Borg and Dienes 1988). The defect reactions proceed as a function of time, but the effect of temperature has long been known to play the most significant role (e.g., Haack 1977). Increases in temperature accelerate the process and, defects will be eliminated until some (quasi-) equilibrium state is established. The overall process of defect elimination is termed annealing (Kelly 1966).

Because of the observed relationship between time and temperature, fission track annealing data have traditionally been displayed on a variation of an Arrhenius plot (e.g., Haack 1972, Laslett et al. 1987) using Equation (5).

$$t = A \exp\left(\frac{E}{kT}\right) \tag{5}$$

Where t is time, A is a mineral specific constant, E is activation energy and T is absolute temperature, with k being Boltzmann's constant. Early researchers (Fleischer et al. 1965a, Wagner 1968, Naeser and Faul 1969, Wagner and Reimer 1972, Nagpaul et al. 1974) noted that annealing data could be represented as iso-density contours (for track density data) or alternatively iso-length contours (for track length data) on such a plot (Fig. 14).



Figure 13. Schematic representation of some important diffusion species in ionic crystals, including cation and anion vacancies, cation and anion divacancies, cation-anion divacancy, allovalent cations (e.g., REE). These are by no means all the likely defect species but illustrate the complex kinetics involved (modified after Borg and Dienes 1988).



Figure 14 — caption opposite page. $\rightarrow \rightarrow$

The Arrhenius equation describes the kinetics of a reaction process but there has been some debate about how to establish the relevant activation energies. Gold et al. (1981) argued that track density measurements were unrelated to the rate constants for the underlying reaction. While Gold et al. (1981) accepted the empirical nature and usefulness of the derived values, they concluded that these parameters were in fact "physically meaningless". The use of the Arrhenius equation, however, gained some justification when Goswami et al. (1984) established a series of analytical equations for the quantitative assessment of variable-temperature track annealing. Goswami et al. (1984) explicitly assumed the validity of the Arrhenius equation in dealing with track annealing data, while Green et al. (1988) presented detailed arguments to refute the interpretation of first-order reaction kinetics for either the shortening of tracks or the reduction of track density. Green et al. argued that while the fundamental process of defect transport in the solid may be described as first-order, the diffusion process becomes extraordinarily complicated in an anisotropic, polyatomic crystal with a spectrum of defect species.

Problems in track measurement

Measurement of the individual diffusion parameters that affect fission track annealing is not routinely undertaken. Rather, we are forced to use a proxy, the tubular hole that remains in the crystal after chemical etching of the fission damage trail. As pointed out by Crowley et al. (1991), etching involves not only removal of the damage trail itself, but also an unknown amount of the host crystal. The diameter of the etched track (~1 μ m) is orders of magnitude larger than the unetched latent track (~10 nm) with a consequent loss of chemical and structural detail. Early studies of track annealing were generally based on track densities, since a clear Boltzmann-law relationship between the track density, time and temperature (Eqn. 5) had been recognized (Fleischer and Price 1964, Naeser and Faul 1969, Haack 1972).

However, track density measurements (e.g., Bertel and Märk 1983) were insensitive to the subtle variations in track length that occur, particularly in samples with complex thermal histories, so that valuable information that may assist with interpretation was lost. And, although a direct relationship between length and density distributions was observed, there was considerable interest in the form of that relationship (Green 1988). Difficulties of inter-laboratory comparison of track density data also may have inhibited developments in fission track modeling (Green et al. 1988). As the method evolved, however, it became clear that confined track lengths provided greater precision in constraining annealing processes and were more amenable to modeling than track densities (Gleadow et al. 1983, 1986; Crowley 1985, Green et al. 1986). As a consequence, there has been widespread acceptance of the utility of combined track length and density measurements for fission track studies. Nevertheless, the track density method continues to find application in some specialized research fields (Carpéna 1998).

The nature of size distributions in etched track lengths is further complicated by the difficulty in determining the dimensions of unetched, or latent tracks. While there were some assumptions about the shape of a latent fission track (see Carlson 1990), their geometry has proven to be notoriously difficult to determine (Kobetich and Katz 1968). In a classic case of the act of observation modifying the observed property—radiolytic annealing was observed in apatite when samples were exposed to the electron beams

Figure 14. Illustration of the extrapolation involved from laboratory scale data (Green et al. 1985) to the geological scale (i.e., months to millions of years). In this case the iso-length contours are fitted using the fanning model of Laslett et al. (1987). Extrapolations of this order magnify the differences between each of the annealing models of Laslett et al. (1987), Carlson (1990) and Crowley (1991). Adapted from Laslett et al. (1987).

applied in TEM (Silk and Barnes 1959, Paul and Fitzgerald 1992) and microprobe analysis (Stormer et al. 1993). There has also been debate regarding the relationship of defect distribution to latent track geometry (Dartyge et al. 1981, Albrecht et al. 1982, 1985; Villa et al. 2000). Even the absolute, initial length of the latent tracks in apatite was (and remains) problematic, partly due to the very process of annealing. Fission fragment ranges have been calculated using range-energy codes (see Henk and Benton 1967, Green 1980, Crowley 1985), which produce an approximately Gaussian distribution. Ranges generated using the Ziegler et al. (1985) "SRIM" package, result in a negatively skewed distribution of fragment ranges.

In an unusual annealing experiment, Donelick et al. (1990) addressed the widespread observation of earlier researchers (e.g., Wagner and Storzer 1970, Bertel et al. 1977, Green 1980, Gleadow et al. 1986) that natural (spontaneous) fission tracks are always shorter than laboratory-induced tracks. Donelick et al. (1990) conducted a series of rapid irradiation and etching experiments and showed that an initial phase of track fading occurs on a remarkably short timescale, even at room temperature. This rapid shortening impacts on the common perception that tracks are completely stable at low temperatures (Naeser 1979a). A shortening of ~0.5 μ m took place at 23°C over three weeks following irradiation, but beyond this time, additional track length shortening was undetectable. Issues of precisely what is a fission track and what are its fundamental, measurable properties are not the only ones facing researchers. The host mineral, apatite, presents us with a number of complexities that have challenged fission track researchers.

Compositional effects

Gleadow and Duddy (1981b) observed variability in the annealing properties of individual apatites from deep wells in the Otway Basin that they attributed to compositional differences. Subsequently, Green et al. (1985) clearly demonstrated a preferential retention of tracks in chlorine-rich apatites relative to fluorine-rich (Fig. 15). These effects have now been widely observed in sedimentary rocks, where the variable provenance of detrital grains contributes to inter-sample variation (Burtner et al. 1994, Corrigan 1993, Stockli et al. 2001), as well as igneous rocks, (e.g., O'Sullivan and Parrish 1995). While fluorine-rich apatites (such as Durango) typically show complete annealing of natural geological samples at temperatures of 90-100°C, chlorine-rich samples are characterized by an increase of the total annealing temperature to around 110-150°C (Burtner et al. 1994).



Figure 15. Variation of individual grain age with chlorine concentration in a single rock sample of volcanogenic sandstone from the Otway Basin in southern Victoria, Australia. The sample was recovered from a depth of 2585 m in the Flaxmans-1 well, where the current temperature is 92°C. The depositional age of the sandstone is indicated by a horizontal line, and the chlorine concentration of the Durango apatite is shown as a vertical dotted line, for comparison. High-chlorine grains resist annealing and thus record an older age than fluorine-rich grains. Cl concentrations are expressed as wt % and as number of atoms per Ca₁₀(PO₄)₆(F,OH,Cl)₂ molecule. Modified after Green et al. (1985).



Figure 16. Plot of apatite fission track age against chlorine concentration for apatite grains in a suite of samples from the Stillwater Complex, Montana. Apatites from these rocks exhibit an unusually wide range of chlorine concentrations. A positive correlation is observed between fission track age and chlorine concentration up to about 4% Cl, reflecting an increasing resistance of the apatite grains to annealing. The most chlorine-rich apatites (~6-7.6 wt % Cl), however reverse this trend and generally yield late Cretaceous-early Tertiary apatite fission track ages similar to those for fluorine-rich apatites. The youngest ages are considered to date relatively rapid regional cooling related to the Laramide Orogeny, the approximate age of which is indicated. The reversal in trend is attributed to a change in apatite fission track annealing behavior accompanying the transition from hexagonal to monoclinic symmetry in the most chlorine-rich apatites. Scales as for Figure 15. Modified after Kohn et al. (2002a).

At temperatures below that of total annealing, chlorine-rich grains may retain more short track lengths (and thus have a shorter mean track length) than fluorine-rich grains experiencing the same thermal history. Thus chlorine-rich grains typically record older apparent ages despite experiencing the same thermal history. An example of this effect in apatites with an exceptional range of halogen compositional variation (sometimes at the hand-specimen level) is reported by Kohn et al. (2002a) from the mafic Archaean Stillwater Complex, Montana (Fig. 16). This compositional dependence response has two important implications. First, samples, particularly sedimentary ones rich in detrital apatite, should be analyzed for halide content to avoid misinterpretation of the ages and thermal history. Second, once the relative annealing kinetics are understood, the added complexity of multi-compositional grains provides the analyst with the power of additional thermochronometers contained in a single sample. Measurements of apatite chlorine content have become largely routine, and a variety of analytical approaches are used (Stormer et al. 1993, Sidall and Hurford 1998). Although the rare earth elements are a significant trace constituent of apatite (Roeder et al. 1987, Hughes et al. 1991b) their influence on annealing processes has been little studied (but see Hurford et al. 2000).

Crystallographic effects

The structural anisotropy of apatite (Sudarsanan and Young 1978, Hughes et al. 1989, 1990) affects the annealing properties of tracks lying in different crystallographic

orientations (Green and Durrani 1977). It appears that the clearly defined channels (notably the anion columns—see Hughes et al. 1990) parallel to the *c*-axis in the crystal structure favor transport of diffusing species (Fig. 17). This results in more rapid annealing of tracks orthogonal to this axis (Green and Durrani 1977). In contrast, tracks oriented parallel to the *c*-axis, appear to be influenced by a less favorable diffusive transport across the crystal lattice, and hence shrink more slowly. The crystal retains a track length distribution spanning these two extremes. As annealing proceeds, the track length anisotropy becomes more obvious (see Wagner and Van den haute 1992). Green et al. (1986) pointed out that observation of horizontal confined tracks on a polished section parallel to the *c*-axis will expose the full angular spectrum of track lengths whereas basal sections will only sample the shortest lengths orthogonal to this axis. Green (1988) subsequently investigated the relationships between anisotropy and length-bias in track shortening and their effects on fission track ages. Later, Donelick and co-workers have significantly expanded the experimental database on crystallographic orientation and anisotropic annealing (Donelick et al. 1990, 1999; Donelick 1991).

Figure 17. Approximate location of F, Cl and OH ions in the anion column parallel to the *c*-axis of apatite(Hughes et al.1989). The ion position shown is for F^- , whereas Cl⁻ and OH⁻ are accommodated by disorder above and below the mirror plane (modified after Sudarsanan and Young 1978).



Numerical annealing models

The development of numerical annealing algorithms has proven to be an enormous analytical breakthrough and is the key to interpretation of results in the majority of recent geological applications. What follows is intended as an overview of the most widely used algorithms, rather than a rigorous assessment of each individual approach. Several other authors provide greater detail and informative discussion on all of the models mentioned here (Gallagher 1995, Willet 1997, Ketcham et al. 1999). By the early 1980s, some classic papers (Wagner and Reimer 1972, Wagner et al. 1977, Naeser 1979b, Gleadow and Duddy 1981b) had already demonstrated the power of fission track analysis in addressing important problems in geology. Interpretation, however, remained a somewhat imprecise art, although several mathematical descriptions of the annealing process had been proposed.

Two contributions (Bertagnolli et al. 1983, Crowley 1985) in particular established formal mathematical treatments to describe the production and shortening of fission tracks in response to thermal history. The approach by Bertagnolli et al. (1983) did not gain wide acceptance, but remained central to much of the work by the French Besançon research group which applied a "convection-type" equation, (Chambaudet et al. 1993, Meillou et al. 1997, Igli et al. 1998). Crowley (1985) used a semi-analytical solution to the problem of track length shortening. He examined several characteristic track-length "signatures" for samples with simple thermal histories and, perhaps more importantly, recognized the applicability of inverse modeling for geological samples. The following year, Green et al. (1986) reported on an extensive laboratory data-set (Fig. 18) that derived from confined track lengths, signaling a major advance in the apatite fission track technique. They also observed that annealing was not accomplished by simple shortening but included a rapid segmentation at high degrees of annealing. The data covered samples heated for times ranging from 20 min to 500 days at temperatures between 95° and 400°C and provided the basis for an empirical, mathematical model published the following year —the widely cited "Laslett et al. (1987) model".



Figure 18. Summary of isochronal laboratory annealing data for confined track lengths in Durango Apatite (Green et al. 1985) for annealing experiments conducted over intervals ranging from 20 min to 30 d. Curves were fitted using the fanning Arrhenius model based on Equation (6a) in the text (adapted from Laslett et al. 1987).

Laslett et al. (1987) based their model on a "fanning Arrhenius relationship" (Fig. 14) between reduced track length $r (= l/l_0$ where l is the measured track length and l_0 is the initial length), log time (t), and inverse absolute temperature (T). They derived the following equation for constant temperature annealing that accounted for 98% of the variation in the observed data:

$$\left[\left\{\left(1-r^{2.7}\right)/2.7\right\}^{0.35}-1\right]/0.35=-4.87+0.000168T\left[\ln(t)+28.12\right]$$
(6)

Subsequent publications by the Melbourne research group illustrated the model's application to variable temperatures (Duddy et al. 1988) and then extrapolated the model to geological time-scales (Green et al. 1989). This collection of papers established this model as the pre-eminent forward model in apatite fission track analysis at the time. As a geological tool, however, its major weakness is that it does not allow for the effect of apatite composition on annealing, being developed entirely on the basis of a single apatite —Durango, a relatively low-chlorine apatite (~0.4 wt % Cl) (see Fig. 15). Hence, application to significantly different compositions will tend to give erroneous paleotemperature estimates. Nevertheless, with the compositional caveat in mind, this model has proved invaluable in understanding low temperature thermal histories in many areas of the world. Laslett and Galbraith (1996) developed an improved version of the Laslett

et al (1987) model with additional parameters. Further adaptations of the Laslett et al. (1987) model have been developed in the commercial environment to address the compositional issue, but remain as yet unpublished.

The Laslett et al. (1987) model was soon followed by additional research on the compositional dependence of annealing. Crowley et al. (1991) conducted over 100 heating experiments that included fluorapatite and Sr-apatite. Their observations suggested that track annealing was consistently anisotropic at all stages of fading, thus allowing tracks of varying orientations to be normalized to a mean track length. They concluded that their annealing results and the data of Green et al. (1986), based on Durango, could be fitted to the following equation:

$$\left[\left\{\left(1-r^{4.3}\right)/4.3\right\}^{0.76}-1\right]/0.76 = -1.508 + \left[\frac{2.076 \times 10^{-5} \ln(t) + 2.143 \times 10^{-4}}{1/T - 9.967 \times 10^{-4}}\right]$$
(7)

Annealing models with these equations lie on a fanning-linear Arrhenius-type plot similar to that of the Laslett et al. (1987) model. The Crowley et al. (1991) model appeared to accommodate the composition problem, in that a near end-member fluorapatite was observed to anneal more readily than Durango (0.4 wt % Cl). However, on extrapolation to geological time-scales this particular model actually predicts fluorine-rich apatite to be more resistant than chlorine-rich apatite (Gallagher 1995). Crowley et al. (1991) interpreted the observed increase in resistance to annealing that occurs during track fading as the prime cause of the fanning form of the "Arrhenius-type" plot (Fig. 14). The fanning results from steepening of the iso-annealing contours and implies an increase in "activation energy" (Gold et al. 1981), which has been interpreted as a type of anneal "hardening".

Only one serious attempt has been made to describe fission track annealing by a physical kinetic model based on atomic-scale mechanisms (Carlson 1990). In essence, Carlson suggested a mechanism where short-range atomic motion caused radial shrinkage of the fission track. Using a simplified track geometry, the change in radial defect distribution could be directly related to a concomitant reduction in track length. The model incorporated parameters for defect distribution, activation energy of the process, a rate constant as well as a parameter controlling the proportion of shortened tracks that suffer segmentation. The kinetic model established the following primary constitutive equation for defect elimination:

$$\frac{\mathrm{d}N}{\mathrm{d}t} = -c \left(\frac{\mathrm{k}T(t)}{\mathrm{h}}\right) \exp\left(\frac{-Q}{\mathrm{R}T(t)}\right) \tag{8a}$$

Here N is the number of defects, t is time, and c is an empirical rate constant. The constants k, h and R are Boltzmann's, Plank's and the gas constant respectively. The term Q is derived from published experimental data, as are the terms A and n in the following equation (Eqn. 8b) where τ is a dummy variable of integration over time and l_o is initial track length (Carlson 1990). When Equation 8a above is combined with the equations describing an approximately Gaussian radial defect distribution, and a function linking axial reduction to varying radius, the resulting equation is:

$$r = 1 - \frac{A}{l_0} \left(\frac{k}{h}\right)^n \left[\int_0^1 T(\tau) \exp\left(\frac{-Q}{RT(\tau)}\right) d\tau\right]^n$$
(8b)

The model attracted considerable criticism and discussion (Crowley 1993a, Carlson 1993a, Green et al. 1993, Carlson 1993b) focusing on two key areas of the model. Firstly, both took issue with the validity of the physical model and its mechanisms. It was argued

that the proposed structure was not based on available physical evidence (Green et al. 1993) and that the mechanism and kinetics of the defect elimination were implausible (Crowley 1993a). Secondly, both dismissed Carlson's (1990) model predictions as having an inadequate fit with the laboratory data sets.

Nevertheless, Carlson's essentially semi-empirical method has gained acceptance, and has been used as the basis for further developments such as the "multi-kinetic" model of Ketcham et al. (1999). This paper is one of three (Carlson et al. 1999, Donelick et al. 1999, Ketcham et al. 1999) that have addressed many of the earlier criticisms of the Carlson (1990) model. This research group has also produced a substantial annealing data-set of mixed-compositional apatites and established a model to deal with crystal-lographic effects, both of which have been incorporated into their full annealing model.

Modeling at an atomic level

Although the empirical models do not provide significant insight into the fundamental processes that occur during track formation and annealing, some indication of the types of mechanisms can be gleaned from basic observations for other materials. For example, one can infer from equations for energy loss phenomena of high-energy particles in solids that the number of displaced ions per unit of track length formed is a function of stopping power of the projectile in the solid and the ionization potential of the target atoms (see Vineyard 1976, Ziegler et al. 1985, Chadderton 1988, Spohr 1990). In a "displacement-spike" or "coulomb-explosion" model of track formation (Fleischer et al. 1965b) (Fig. 2), this implies that lattice atoms with a low ionization potential (e.g., chlorine) will be readily ionized and displaced into the surrounding lattice. In contrast, fluorine has a higher ionization potential and is therefore less readily ionized. The overall result predicted is a larger track diameter in high-chlorine apatite than in fluorine-rich specimens. This is consistent with the observations based on the bulk etch rates in routine fission track analysis (Carlson et al. 1999). Track diameters have been used as a proxy for halide content with the "etch figure" method where the mean etch pit diameter is measured parallel to the crystal *c*-axis (Burtner et al. 1994, Donelick et al. 1999, Stockli et al. 2001).

James and Durrani (1988) suggested that in addition to having a spatial distribution, defects, such as those in Figure 13, may also be represented by a "potential-energy-well distribution". This concept has a direct bearing on the energy subsequently required to mobilize the defect. If the bulk of these displaced ions cannot immediately be accommodated in their "normal" lattice sites, they will lodge in the matrix as "selfinterstitials". Several processes are then likely to occur. Those within 5-10 lattice spacings may spontaneously recombine with their corresponding vacancy (Borg and Dienes 1988)-either in the track core or a lattice vacancy caused by the knock-on event (Itoh and Tanimura 1986). For interstitials beyond the strain field of such vacancies, the large size of anion interstitials makes them quite unstable. Anions tend to be the most mobile and have the lowest migration or activation energy. Cation interstitials, in contrast, are smaller and will have slightly higher activation energies. Interstitial ions typically have much lower activation energies than vacancies and are more mobile. Since in fission tracks, such processes involve significant movement through the lattice, the transport process (diffusion) is likely to be the rate controlling parameter (Mrowec 1980). Collectively, these diffusion processes contribute to the relative stability of the vacancydominated track in insulators when compared to metals (Chadderton 1988).

The kinetics of clusters further enhances the stability of tracks in insulators (Dartyge et al. 1981). Trapping of various defects by allovalent substitutional atoms is also highly likely and will result in an increase in apparent activation energies for participating ions due to sequential trapping and de-trapping reactions preceding a final recombination.

Given the abundance of trivalent, rare-earth elements commonly substituting for divalent calcium in the apatite (Roeder et al. 1987, Hughes et al. 1991a,b), the contribution of this process cannot be dismissed as insignificant. These reactions are further complicated by the crystal structure (Sudarsanan and Young 1978) with its characteristic anion channels parallel to the *c*-axis (Fig. 17). A less prominent cation channel is also present. Currently, atomic scale approaches remain theoretical, but there is little doubt that advances in this field could dramatically improve our understanding of the fundamental processes involved in track annealing in apatite.

Thermal history reconstruction and inversion modeling

Annealing models, however parameterized, are designed to simulate nature. In these models, time and temperature represent the input, and outputs are in the form of track length distributions and apparent age. Forward modeling enables one to predict the resulting fission track parameters from any hypothetical thermal history that can then be compared with the actual observations on geological samples. Forward modeling has been used initially as a means of checking annealing models against well-constrained geological examples (e.g., Green et al. 1989) and as a guide to interpretation (e.g., Willett 1992) of real samples. This approach, however, is a very inefficient means of finding solutions for unknown (or poorly constrained) samples. What is required is a method for extracting the actual, or at least a most probable, thermal histories directly from the observed data by inversion modeling.

One favored method involves the use of a 'genetic' algorithm (Gallagher and Sambridge 1994) to search time-temperature space for solutions that reproduce the observed data with the least amount of variance. Since the solutions matching the observed data are not unique, a large number (typically thousands) of possible histories must be tested and an assessment of likelihood or probability is made for each. Solutions can be more tightly constrained by incorporating additional geological and thermal information. Because track annealing is a unidirectional process, high temperatures have the effect of resetting (or at least significantly modifying) the track length distributions. As a result, inversion modeling only provides robust thermal histories subsequent to maximum heating (or burial) of the sample. Prior to such heating the solutions are only poorly constrained and certainly unreliable.

Because the annealing process is mathematically non-linear, solutions cannot be determined by routine least-squares optimization procedures, and Monte-Carlo approaches to sampling the time-temperature parameter space are used (Corrigan 1991, Lutz and Omar 1991, Gallagher 1995). Corrigan (1991) used a stochastic optimization, whereas Lutz and Omar (1991) applied an iterative method called the 'downhill simplex method.' Gallagher's (1995) inversion method relied on a stochastic search using a genetic algorithm to improve the number of good data-fitting solutions as illustrated in Figure 19. More recently, Willett (1997) used a controlled random search algorithm in which the solutions are similarly assessed on the quality of fit with the observed data. A wealth of software has been developed to automate the inversion modeling described here (Crowley 1993b, Gallagher 1995, Issler 1996, Ketcham et al. 2000). Typically the computer code written for inversion models allows the user the option of choosing the preferred annealing model (such as described above) as well as accommodating compositional variations.

GEOLOGICAL APPLICATIONS OF APATITE FISSION TRACK ANALYSIS

Apatite fission track analysis has been applied to a broad range of geological problems (e.g., Wagner and Van den haute 1992, Ravenhurst and Donelick 1992, Brown et al. 1994, Andriessen 1995, Gallagher et al. 1998, Gleadow and Brown 2000, Dumitru



Figure 19. Output typical of an inversion model (searching time-temperature space using a Monte-Carlo approach). The upper frame shows model-generated histories that are each tested against the observed data (track length distribution and fission track age). Various algorithms are used to iteratively improve the model "fit" against the data. The lower frame illustrates a graphical output of the model "best fit" length-distribution compared to the measured track lengths. (generated using MonteTrax; based on Gallagher 1995).

2000, Gunnell 2000. Here, we present an overview of the mainstream applications and cite representative literature related to each topic. A flow chart showing the sequence of steps and inputs used to derive different geologically useful outputs from apatite fission track data and their linkages to various applications is summarized in Figure 20.

As indicated in the preceding discussion, apatite fission track data in most cases give rise to apparent ages, which reflect regional patterns of cooling, rather than the original formation ages of the rocks involved. Mostly, the apparent ages obtained are 'mixed' ages which reflect more than one component of the thermal history and only in a relatively limited number of circumstances do they directly date a particular episode or discrete geological event (Fig. 9).

Absolute dating

Absolute age dating using apatite is only possible in very restricted circumstances and has been applied to volcanic and shallow intrusive rocks, meteorite impact events and contact metamorphism. The fundamental requirement is that samples cooled rapidly and subsequently remained thermally 'undisturbed' and close to or at the surface. Due to its relatively low uranium content and restricted occurrence, apatite dating of Quaternary **Figure 20 (opposite page).** Flow chart showing the sequence of steps and possible inputs which can be used to derive geologically useful output parameters from apatite fission track data. Outputs such as regional thermal history, denudation and paleotopography may also be displayed as images for different time slices or as movies. Sources of error involved are cumulative so that uncertainties increase with each step away from the primary apatite fission track data. Linkages of outputs to various geological applications are also shown (modified after Kohn et al. 2002b). $\rightarrow \rightarrow$

volcanic rocks is not commonly used, compared to the more ubiquitous zircon and/or glass. Zircon is most suitable in this time range, because of its relatively high uranium content and resistance to weathering. When used, apatite dating of volcanics is usually carried out together with fission track dating of coexisting zircon and/or glass or other radiometric dating methods (e.g., ⁴⁰Ar/³⁹Ar). Apatite from three rapidly cooled igneous rocks, the Fish Canyon Tuff (Naeser et al. 1981, Hurford and Hammerschmidt 1985), the Cerro de Mercado Martite (the 'Durango' apatite, Naeser and Fleischer 1975), and the Mt Dromedary Igneous Complex (Green 1985), are commonly used as standards to calibrate fission track dating (Hurford 1990). Apatite has also been used in conjunction with other minerals to constrain ages of kimberlite and diatreme emplacement (e.g., Naeser 1971, Brookins and Naeser 1971). An early comparative study of apatite fission track ages and K/Ar whole rock and plagioclase ages from early Miocene seamounts in the Gulf of Alaska (Turner et al. 1973) indicated concordant ages (within errors quoted), suggesting little or no track annealing in the ocean floor environment. However, in this example the apatite ages are not robust, because their 2σ errors are more than three times those obtained by K-Ar and no track length measurements are reported.

The complete or partial resetting of apatite fission track clocks in target rocks by shock-wave heating has been used to constrain the timing of meteorite impacts events (e.g., Wagner 1977, Storzer and Wagner 1977, Hartung et al. 1986, Omar et al. 1987 and Kohn et al. 1995).

Calk and Naeser (1973) demonstrated the possibility of dating shallow intrusions indirectly. A late Cretaceous quartz monzonite body in Yosemite National Park, California, was intruded by a high level basaltic plug. Apatite and sphene fission track dates from the quartz monzonite within 5 feet of the contact with the basalt yield concordant late Miocene ages. These 'country rock' ages result from their total thermal resetting as a consequence of their proximity to the basalt intrusion, which they date by proxy. A further example of apatite dating of a discrete thermal event is the "Mottled Zone" in Israel and Jordan, formed during high-T, low-P metamorphism by near surface combustion of organic matter related to the development of the Dead Sea Transform system (Kolodny et al. 1971).

Apatite fission-track crustal profiles

The sensitivity of fission tracks in apatite to the relatively low temperatures characteristic of the upper few kilometers of the crust (~0-120°C), and the consequent patterns of track length distribution and apparent fission track age with depth that evolve within the crust, provide an unparalleled tool for studying the long-term thermal evolution of the Earth's crust. The detailed form of these fission track profiles reflects the distribution of temperature within the shallow crust and its variation through time. The profiles are therefore characteristic of the style of thermal history experienced by the crustal section. A sequence of samples representing a range of depths within the crust, such as a suite of samples collected from deep drill holes (e.g., the Otway Basin, Fig. 11), or from outcropping rocks collected over a range of surface elevations, can thus be used to document the vertical pattern of fission track parameters within the upper crust. These



fission track profiles can then be used to reconstruct the thermal history for the sampled section and thus place quantitative constraints on its thermal history. The potential of using apatite fission track profiles in this manner was first documented in the European Alps by Wagner and Reimer (1972), and Wagner et al. (1977), and subsequently in a profile from the Eielson drill-hole in Alaska (Naeser 1979b).

In regions of the crust that have not been disrupted by tectonic movements and where rates of erosion have been low (\sim 30 m Myr⁻¹) (Gleadow 1990, Brown et al. 1994) for a prolonged period ($>10^6$ years), such as cratonic interiors, or where progressive burial has been occurring, as in sedimentary basins, the typical form of the fission track crustal profile is controlled primarily by the progressive increase of temperature with depth. The pattern produced by this situation of continuous residence at constant or gradually increasing temperature is one of progressive reduction in mean track length and hence fission track age with depth (e.g., Fig. 11). The region of most rapid reduction in apparent fission track age and track length is described as the partial annealing zone (PAZ), typically between about 60° and 110°C.

More complicated thermal histories will produce correspondingly more complex apatite age profiles. If there is a rapid increase in the rate of erosion after a prolonged period of geomorphic stability, then the base of the existing concave-up apparent age profile will be shifted upwards towards the new mean topographic surface as denudation proceeds (e.g., Gleadow and Brown 2000). Apatite samples that were at temperatures > ~110°C prior to the acceleration in denudation will have accumulated no fission tracks and hence have zero apparent ages up to that point. On the initiation of cooling produced by the accelerated erosion these samples will begin to retain fission tracks below ~110°C and a new apparent age profile will begin to develop below the earlier profile as shown in Figure 21. If the amount of crust removed during this episode is of the order of a few kilometers then part of the earlier apparent age profile may be preserved within the upper sections of the new topographic relief. The transition from the earlier upper profile to the

Figure 21. Modeled fission track age and mean track length profiles in apatite from a hypothetical two-stage cooling history incorporating a distinct cooling episode at time t. The cooling paths of samples at successively increasing depth in the final profile are shown in (a). The resulting profiles of both apatite age and track length (b) are distinctive and show a characteristic inflexion point or break in slope, the age of which closely approximates the time of onset of the cooling event. The break in slope represents the position which was at the base of the apatite fission track annealing zone prior to the onset of rapid cooling. The outlined rectangle in (b) represents the segment in the example shown in Figure 22. After Gleadow and Brown (2000).

new lower profile will be marked by a pronounced inflection (Fig. 21). The location of this inflection marks the depth at which the apparent apatite age was reduced to zero (i.e., the depth of the ~110°C paleo-isotherm) prior to the increase in erosion rate, and the corresponding age approximates the time at which the acceleration in erosion took place (Fig 21). Such inflection points have now been observed in vertical profiles sampled over significant topographic relief in various parts of the world (e.g., Gleadow and Fitzgerald 1987, Fitzgerald and Gleadow 1988, Fitzgerald et al. 1995, Foster and Gleadow 1996, Kohn et al. 1999) and are recognized as a feature of crustal blocks that have undergone episodic kilometer-scale denudation. An example from Denali in the Alaska Range is shown in Figure 22.

Figure 22. An apatite fission track age-elevation profile from Denali in the Alaska Range of North America. The profile shows the characteristic break in slope separating an upper zone of rapidly increasing age and mixed to bimodal length distributions from a lower steep zone where the age shows little variation and the length distributions are long and narrow. The inflection point at about 6 Ma represents the time of onset of rapid cooling in this profile, presumable related to rapid denudation consequent on uplift of the range. The two numbers on each length histogram represent the mean track length (top) and the standard distribution of the distribution (bottom). Modified after Fitzgerald et al. (1995).

The form of the new apparent age profile that develops below this inflection will depend on both the rate and duration of the accelerated period of denudation, and could be either a linear gradient in apparent age with depth if sufficient section were removed, or possibly a new concave-up PAZ profile. Clearly, it is possible to produce a composite profile with an inflection comprising two linear components with differing age-depth gradients representing a change from a moderate rate of erosion to a significantly higher rate. Interpreting an observed gradient in apparent apatite age therefore requires discriminating between prolonged annealing at constant temperature (very low rate or no erosion) and continuous erosion at some constant rate. Resolving these two interpre-

tations of an age gradient is obviously a crucial step in arriving at a meaningful interpretation of any apatite fission track crustal profile and depends critically on the information represented by the distribution of individual track lengths of a sample.

Ore deposits

Several apatite studies have aided in deciphering the time-space relationships between igneous and/or hydrothermal events and their associated mineralization (Arne 1992). These studies, also employing other radiometric dating techniques, have been mainly applied to precious and base metal deposits environments in the western USA (e.g., Banks and Stuckless 1973, Ludwig et al. 1981, Cunningham et al. 1984, Lipman et al. 1976). Paleothermal anomalies, identified from apatite cooling patterns beneath mineralized rocks in Colorado, were postulated to result from the presence of buried stocks (Naeser et al. 1980, Cunningham and Barton 1984, Beaty et al. 1987). Identification of the anomaly reported by Naeser and co-workers led to the discovery of a major molybdenum ore body (Cunningham et al. 1987).

Stratigraphic dating and provenance

Gleadow and Duddy (1981a) demonstrated the utilization of apatite to constrain the maximum age of strata. They showed that most of the fluviatile sediments forming the Early Cretaceous Otway Group deposited in the Otway Basin, a rift graben in southeastern Australia, comprised volcanogenic detritus derived from contemporaneous volcanism to the east. In outcrop sections of the Otway Group, sphene, zircon and apatite fission track ages, three minerals with greatly different closure temperatures, were concordant within error, indicating that the outcrop sections have never been significantly heated since deposition. Hence, the sediments represent the original ages of the detritus and provide maximum ages for the sedimentary host. In general however, because of its low temperature for fission track retention, apatite is not as suitable as zircon for dating source ages for provenance studies (Storzer and Wagner 1982, Hurford and Carter 1991, Carter 1999). Carter (1999) emphasized that detrital apatite fission track applications should aim to improve the resolution of the temporal relationships between source evolution and sedimentation in adjacent basins. Brookins and Naeser (1971), Ross et al. (1976), Turner et al. (1983) and Carter et al. (1995) demonstrate other approaches using apatite fission track data for constraining stratigraphic interpretations.

Sedimentary basins

A widespread application of apatite thermal history studies is in sedimentary basins, particularly for the interpretation and quantitative modeling of thermal histories, and hydrocarbon resource evaluation. Naeser (1979b) described the expected apatite age versus depth trends for different basinal thermal histories, and presented the earliest discussion of the potential of this approach. Subsequent studies in the Otway Basin (e.g., Gleadow and Duddy 1981b, Gleadow et al. 1983, Green et al. 1989, Mitchell 1997) and other basins (e.g., Briggs et al. 1981, Storzer and Selo 1984, Dumitru 1988, Naeser et al. 1989, Seward 1989, Naeser et al. 1990, Duane and Brown 1991, Naeser 1993, Issler at al. 1990, Kamp and Green 1990, Crowley 1991, Steckler et al. 1993, Blackmer et al. 1994, Duddy et al. 1994, Gallagher et al. 1994a, Green et al. 1995, Hill et al. 1995, O'Sullivan 1996, Giles and Indrelid 1998) have demonstrated how apatite studies may provide unique constraints on paleo-geothermal gradients, estimates of maximum paleotemperatures and their timing, basin inversion and erosion, fluid flow, and mechanisms of basin formation. The information may also be used to assess hydrocarbon potential that has led to increased commercial application of the fission track method. Several basin studies have also used a combined, multi-method approach for thermal history modeling using a variety of thermal indicators such as apatite fission track analysis, vitrinite reflectance, fluid inclusions, clay mineralogy and argon radiometric data (e.g., Feinstein et al. 1989, Kohn et al. 1990, Bray et al. 1992, Grist et al 1992, Arne and Zentilli 1994, Burtner et al. 1994, Kamp et al. 1996, Tseng et al. 1996, Zhao et al. 1996, Mitchell 1997, Pagel et al. 1997, Kohn et al. 1997, Parnell et al. 1999, Marshallsea et al. 2000, Mathiesen et al. 2000, Hu et al. 2001, Osadetz et al. 2002). Apatite studies related to carbonate-hosted Mississippi Valley-type (MVT) Pb-Zn ore deposits (Arne et al. 1990, Arne 1992) suggest that such mineralization is associated with broad regional heating rather than with discrete hot pulses of metalliferous brines migrating through sedimentary basins, as previously envisioned.

Orogenic belts

One of the earliest applications exploited by the temperature-sensitive nature of the apatite fission track system was for the reconstruction of thermal histories in mountain belts. Wagner (1968) and Wagner and Reimer (1972) first demonstrated the potential of the fission track method for studying tectonic problems, thus laying the foundation for expanding the scope from an 'age determination' approach alone to a unique thermotectonic tool. Quantitative information arising from fission track studies relate to the postorogenic cooling history, and potentially provide estimates of the timing, magnitude and rate of erosional and tectonic denudation. Some early apatite studies in mountain belts used age versus elevation plots to directly derive denudation and uplift rates, but the relationship to the fission track parameters may be been oversimplified (e.g., Summerfield and Brown 1998, Gallagher et al. 1998). Some complicating factors are due to: (1) inclusion of samples containing tracks which accumulated prior to rapid cooling (i.e., from a paleo PAZ), (e.g., Roberts and Burbank 1993), (2) perturbation of crustal isotherms during denudation and the development of the topography (e.g., Parrish 1983, 1985, Kohn et al. 1984, Stüwe et al. 1994, Brown and Summerfield 1997), (3) postcooling rotation or folding of the sampled rocks, complicating interpretation of data from vertical profiles (e.g., Johnson 1997, Rahn et al. 1997), and (4) lateral particle motion parallel to isotherms, rather than vertical transport perpendicular to isotherms (e.g., Willett et al. 1993, Batt and Brandon 2002). Apatite studies have been reported from most of the world's young orogenic belts including: the Alps (e.g., Schaer et al. 1975, Wagner et al. 1977, 1979; Hurford 1986, 1991; Schlunegger and Willett 1999), the Tatra Mountains (Burchart 1972), Pyrenees region (Yelland 1990, Fitzgerald et al. 1999, de Bruijne and Andriessen 2002), Calabrian Arc (Thomson 1994), Ural Mountains (Seward et al. 1997, Leech and Stockli 2000, Himalayas (Sharma et al. 1980, Zeitler et al. 1982, Zeitler 1985, Sorkhabi 1993, Foster et al. 1994, Sobel and Dumitru 1997), Rocky Mountains (Naeser 1979a, Bryant and Naeser 1980, Roberts and Burbank 1993), Taiwan (Liu 1982), Western North America (Harrison et al. 1979, Parrish 1983, Plafker et al. 1991, O'Sullivan et al. 1993, 1997; O'Sullivan and Parrish 1995, Fitzgerald et al. 1995, Brandon et al. 1998, Dumitru 1991), Andes (Nelson 1982, Crough 1983, Kohn et al. 1984, Shagam et al. 1984, Benjamin et al. 1987, Jordan et al. 1989, Coughlin et al. 1999, Spikings et al. 2000, Thomson et al. 2001); New Zealand Alps (White and Green 1986, Kamp et al. 1989, Tippett and Kamp 1993, Batt et al. 1999, 2002) and Papua-New Guinea (Hill and Gleadow 1989, Hill and Raza 1999).

Non-orogenic settings

Passive continental margins. Apatite fission track thermochronology also reveals the pattern and chronology of denudation in relation to the development of topography and landscape features in the on-shore region of rifted continental margins. Such data contribute to an improved understanding of the evolution of lithospheric thermal and strength distribution and the nature of rift-flank tectonic uplift associated with extension and surface processes (van der Beek et al. 1994, 1995). In the case of passive margins,

the time-space relationship of onshore denudation is also important for calculating sediment volumes in adjacent offshore basins, which may have formed during extension. Apatite track studies on detrital grains and basement clasts in orogenic sediments (Wagner et al. 1979, Garver et al. 1999) and in offshore sediments (Clift et al. 1996, Gallagher and Brown 1999b) have been employed to reconstruct the exhumation histories of nearby mountainous areas and adjacent onshore passive margins, respectively. Onshore studies along rifted continental margins and rift flanks have been reported from southeastern Australia (Gleadow and Lovering 1978, Moore et al. 1986, Dumitru et al. 1991, Foster and Gleadow 1992a, O'Sullivan et al. 1995a, 1996, 1999, 2000b; Kohn et al. 1999), eastern Greenland (Gleadow and Brooks 1979, Thomson et al. 1999a, Hansen 2000, Johnson and Gallagher 2000, Mathiesen et al. 2000), Alaska (Dumitru et al. 1995), Rio Grande rift (May et al. 1994), northern United Kingdom (Green 1986, 2002; Lewis et al. 1992, Green et al. 1999, Thomson et al. 1999b), Baikal rift (van der Beek et al. 1996, van der Beek 1997), southern Norway (Rohrman et al. 1994, 1995), Red Sea-Gulf of Suez (Kohn and Eyal 1981, Bohannon et al. 1989, Omar et al. 1989, Omar and Steckler 1995, Kohn et al. 1997, Menzies et al. 1997), Transantarctic Mountains (Gleadow and Fitzgerald 1986, Fitzgerald et al. 1986, Fitzgerald 1994, Schäfer and Olesch 1998, Lisker 2002), southern Africa and southeast Brazil (Brown et al. 1990, Gallagher et al. 1994b, Gallagher and Brown 1997, 1999a,b; Brown et al. 2000, Cockburn et al. 2000, Raab et al 2002) and East Africa (Foster and Gleadow 1992b, 1993, 1996; Noble et al. 1997). Quantification of denudation history arising from apatite studies has been used to constrain models for the development of major escarpments formed during continental break-up (Gallagher and Brown 1999a, Cockburn et al. 2000). Blythe and Kleinspehn (1998) and Winkler et al. (1999) have also evaluated the possible role of climatically driven erosion as a component of the exhumation interpreted from apatite data. O'Sullivan and Brown (1998) explained Miocene cooling observed in well samples along the northern Alaska coastline as a response to a long-term decrease in the paleo-mean annual surface temperature.

Extensional terranes. In highly extended terranes, cooling recorded below largescale detachment faults and crustal shear zones may result predominantly from normal faulting, i.e., tectonic denudation as opposed to erosional denudation. In extensional settings apatite fission track used together with other thermo-chronological methods may provide unique information on: (i) the timing of rapid crustal extension, (ii) rates of cooling, (iii) average slip rates on detachment faults, (iv) paleo-geothermal gradients prior to and following rapid extension, and (v) initial dips of detachment faults and tilted hanging wall blocks. Examples of apatite fission track studies in extensional settings include: Western USA (Foster et al. 1991, 1993; Fitzgerald et al. 1991, 1993; Dokka 1993, John and Foster 1993, Howard and Foster 1996, Foster and John 1999, Miller et al. 1999, Pease et al. 1999, Fayon et al. 2000, Stockli et al. 2001, Foster and Raza 2002), Gulf of California region (Axen et al. 2000, Fletcher et al. 2000), Greece (Thomson et al. 1998a,b; Hejl et al. 2002) and Papua-New Guinea (Baldwin et al. 1993).

Cratons. Ancient crystalline terranes, forming extensive cratons and large tracts of continental interiors, are generally viewed as tectonically and isostatically stable, and resistant to internal deformation. A growing body of evidence however, based on reconnaissance apatite studies of different cratons and Precambrian blocks, has revealed discrete, regional episodes of Phanerozoic km-scale crustal erosion of these ancient terrains (Crowley et al. 1986, 1991; Zeck et al. 1988, Brown et al. 1990, Noble et al. 1997, Spikings et al. 1997, Harman et al. 1998, Mitchell et al. 1998, Cederbom et al. 2000, Cederbom 2001, Gibson and Stüwe 2000, O'Sullivan et al. 2000a, Kohn et al. 2002b, Osadetz et al. 2002). These surprising results are difficult to reconcile with the widely held view of cratons as being tectonically and erosionally inert but open a

promising avenue of research for further detailing the morpho-tectonic evolution of these ancient terranes.

Fault displacement and reactivation

In crystalline terranes traditional stratigraphic markers that might be used for reconstructing regional structure and tectonic evolution are usually not available. In such environments, apatite data from surface outcrops and deep drillhole samples may be used to characterize paleodepth thermo-chronological markers, in effect an 'invisible' stratigraphic tool. This approach has been used to place unique constraints on the magnitude and timing of relative fault displacement and regional structure (Tagami et al. 1988, Wagner et al. 1989, 1997; Fitzgerald and Gleadow 1990, Dumitru 1991, Foster and Gleadow 1992a,b, 1996; Kohn 1994, O'Sullivan et al. 1995b, 2000a; Coyle et al. 1997, Cox et al. 1998, Kohn et al. 1999), as well as the reactivation history of ancient, fundamental faults or lineaments (Harman et al. 1998, O'Sullivan et al. 1998, 1999; Raab et al. 2002).

Regional and continental-scale thermo-tectonic imaging

Apatite fission track ages rarely reflect the formation ages of the host rocks sampled, and on a regional or continental-scale often show broad spatial variations that reflect their thermal and denudation histories. Further, the form in which these data have been presented have often been difficult for non-specialists to interpret and their implications difficult to visualize. Recently, strategies have been developed for inverting such large data sets into time-temperature solutions, which can be visualized as a series of time slice images and movie sequences, depicting the cooling history of present day surface rocks during their passage through the upper crust (Gleadow et al. 1996, Gallagher and Brown 1999a,b; Gleadow et al. 2002, Kohn et al. 2002b). The data can be further extended and combined with other large-scale data sets, such as digital elevation and heat flow, to image other geologically useful parameters, such as denudation and paleotopography (see Fig. 20). Such images provide an important new perspective on the crustal processes and landscape evolution and permit tectonic and denudation events to be visualized in time and space.

CONCLUSIONS AND FUTURE DIRECTIONS

After some thirty years of investigation and development, fission track analysis of apatite has produced robust methodologies with a wide range application to geochronology and, particularly, to low-temperature thermochronology. The particular sensitivity of apatite fission tracks to the temperatures prevailing in the low-temperature environment of the upper crust has opened new opportunities for the chronological investigation of surface processes and tectonics not accessible to earlier approaches. Apatite fission track analysis has also proved to be of unparalleled utility for paleotemperature reconstruction in sedimentary basin environments with important implications for hydrocarbon exploration. These developments mean that the dominance of apatite as the mineral of choice in fission track analysis is likely to continue.

Despite the substantial progress achieved in techniques, interpretative strategies and modeling over the last twenty years, and an increasing standardization of approach in most laboratories, there is still room for considerable improvement. One limitation of current techniques is the necessarily limited precision of fission track analysis due to the relatively small numbers of tracks counted, and the reliability of track identification dependent to a large degree on the skill and experience of the analyst. While the generally good reproducibility achieved in inter-laboratory comparisons suggests these are not major concerns, there is no fundamental barrier to improved precision through an order of magnitude increase in the number of tracks counted. This situation is not likely to improve using existing manual track counting methods, but eventually, it should be possible to develop fully automated track counting which would remove the present limitations, determined as they are mostly by the endurance of the analyst. Previous attempts at automated track counting by image analysis have not as yet proved practical, but it can be anticipated that this is likely to change over the next five years.

A second area where a significant departure from traditional methods may be at hand is in direct analysis of uranium by, for example, laser-ablation inductively-coupled plasma mass spectrometry (LA-ICPMS). The possibility for the rapid determination of uranium at $\mu g/g$ levels in individual apatite grains would be a radical departure from all previous approaches. Such an approach would make the present use of neutron irradiation unnecessary, and lead to improvements in analytical precision and sample turn-around time, as well as improved laboratory safety by eliminating the minor levels of radioactivity that must currently be managed. Such a method would also eliminate many of the calibration issues that have pre-occupied fission track workers for many years, although, of course, a new set of calibration problems would undoubtedly arise. Direct uranium determination would also open the way for a reinvestigation of monazite for fission track dating, which might have important applications in low-temperature thermochronology.

A third area where significant improvement can be anticipated is in closing the gap between the current generation of purely empirical annealing models and a fundamental understanding of the lattice processes involved in annealing. New models can be anticipated which will relate more closely to the atomic scale diffusion that controls the repair of fission damage in apatite and other minerals. Even without such developments, we can anticipate the emergence of a new generation of well-documented multicompositional apatite annealing models that should significantly improve the reconstruction of thermal histories and enhance interpretation of apatite fission track data.

Many important insights in thermochronology have arisen from the application of apatite fission track analysis, either alone or in combination with other techniques, such as 40 Ar/ 39 Ar dating, which record higher temperature history information. Most fission track studies have produced only limited information on the very-low temperature history (<~60-70°C), characteristic of the shallowest crustal levels, due largely to the uncertainty of kinetic models in this range. This has limited the ability to close the gap between the deeper, subsurface evolution of the rock masses involved, which are reasonably well characterized, and processes acting at surface and near-surface levels. An important technical development in recent years, the advent of (U-Th)/He thermochronometry on apatites (see Farley and Stockli, this volume), provides an exciting opportunity to address this issue directly.

The temperature range of the He apatite partial retention zone (between ~40 and 80°C) records even lower temperature information, and often younger ages, than does apatite fission track thermochronology. Hence, in many cases shallower crustal depths and possibly less structural relief may be required to distinguish among various cooling histories using the apatite (U-Th)/He method. As the partial retention temperature ranges for the two methods partly overlap, apatite He ages will provide quantitative tests of track-length derived thermal models. Comparison of the two methods will thus increasingly be used to validate and crosscheck laboratory calibrations in both systems. Apatite (U-Th)/He thermochronometry is therefore an ideal complement to apatite fission track studies in that it is most sensitive over the temperature range where the apatite fission track system is least sensitive. Future challenges will involve the development of new protocols for the integrated modeling of low temperature histories obtained from both methods. Integration of these methods on both local and regional scales will lead to

the development of new strategies for the time-space visualization and improved understanding of upper crustal and surface processes. The combined application of these two methods of thermochronology to the same apatites has great potential to provide significantly more information than either method alone. For this reason it can be foreseen that the predominance of apatite in low temperature thermochronology is likely to be sustained.

ACKNOWLEDGEMENTS

We are grateful to Günther Wagner, Tony Hurford, Matt Kohn and Cathy Skinner for their extremely helpful reviews that have substantially improved the original version of this manuscript. We also thank our numerous colleagues and students in the Melbourne Fission Track Research Group over many years from whom we have gained much and benefited in our understanding of the many issues covered in this chapter. This work has been supported by the Australian Research Council, the Australian Geodynamics Cooperative Research Centre and the Australian Institute of Nuclear Science and Engineering.

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