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Etching zircon age standards for fission-track analysis

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Abstract

Nineteen laboratories that routinely measure fission-track ages in zircon were surveyed as to their principal methodology used for track revelation using chemical attack and counting procedures. The survey results show the following: (a) researchers in most labs count fission tracks with a optical microscope using at a total magnification between 1250× and 1600× (∼ 80%) with about an equal number using either a dry or oil objective; (b) the majority of laboratories etch zircon with a KOH:NaOH eutectic heated in an oven between temperatures of 210°C and 230°C; (c) age standards in zircon analysis do not have uniformly accepted etch times. Etching times for the widely used 28 Ma Fish Canyon Tuff (FCT) (4–60 h) and the lesser-used 16 Ma Buluk Tuff (13–55 h) vary significantly from lab to lab. Between ∼ 220°C and 230°C, the principal range for etching times for the FCT is between 20 and 30 h, and the mode for the Buluk Tuff is between 30 and 55 h. Three or fewer labs report etching times for the Tardee Rhyolite (22–40 h), the Bishop Tuff (10–46 h), and the Mt. Dromedary Banite (5–24 h). Variation in etching times may result in a bias in U-content which affects counting statistics. If etching is successful, strict criteria must be followed to ensure that the analyst only counts well-etched grains and that all tracks are successfully identified.

Keywords: Fission track; Zircon; Etching; Age standards; Fish Canyon Tuff

1. Introduction

In the last decade, fission-track (FT) analysis using apatite and zircon has seen rapid growth and widespread application in a number of disciplines in the earth sciences. Although FT analysis of apatite is widely used, zircon receives comparatively little attention, which may partly be due to the difficulty of proper track revelation. FTs in zircons have been studied for nearly 40 years. The first published paper to describe sample preparation, etching, and track density determination in zircon crystals for fission-track dating used cleaved (split) crystals immersed in boiling phosphoric acid to reveal tracks (Fleischer et al., 1964). Since the introduction of mounting zircon in Teflon® and then etching with a strong base (KOH:NaOH, NaOH and water, or KOH:NaOH:LiOH), there have been few changes in the basic methodology for handling zircon for routine FT analysis (i.e. Gleadow et al., 1976; Naeser, 1976; Gleadow, 1978; Zuan and Wagner, 1985). By the early 1980s most labs had settled on using a technique where Teflon®-embedded zircons grains were etched in a KOH:NaOH eutectic between ∼ 200°C and 230°C, but a quantitative criteria for track revelation was lacking. In the 1990s, important advances were made by the FT group at Kyoto University in understanding track-length distribution in zircon and specific etching criteria (see, for example, Tagami et al., 1990; Hasebe et al., 1993; Yamada et al., 1995). In this approach, spontaneous tracks in zircon are etched until tracks perpendicular to the *c*-axis are ∼ 1 μm wide (larger for track-length studies and annealed samples, see Hasebe et al., 1993).

Despite the availability of this more quantitative approach to track revelation, many in the FT community encounter difficulties obtaining an optimal etch from sample to sample. This problem is undoubtedly due to sample-to-sample variation in α-damage (combined α-recoil and α-particle damage), which affects the response of zircon to chemical attack. In general, the effect of accumulated α-damage is to change the chemical reactivity of the zircon crystal, resulting in a reduction of etching time. Remarkably, even though

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there is only minor variation in the details of how different laboratories operate, there is a wide variation in the reported etching times for age standards. The implications for such lab-to-lab variation are important: either the system is suited to a wide range in etching condition, or the basic calibration of the zircon FT method is suspect. Ideally, with more uniformity between labs, the FT community may be able to measure consistent, meaningful data from zircon.

As a starting point, a number of laboratories that routinely analyze zircon by the FT method by the external detector method were surveyed as to their methodology. This paper summarizes the results of this poll, concluded by early 2000, and presents interpretations of these data. Finally, some recommendations are made for emerging labs so that they can benefit from the collective experience of the FT community.

2. Survey

Thirty labs were contacted between November 1999 and January 2000. About 19 responses had sufficient information to be included in the tabulated survey (Table 1). Note that these 19 responses represent the majority of labs that routinely date zircon by the FT method (Table 1, Fig. 1). The survey questions focussed on the main factors that may vary between labs: (a) magnification and objective used for counting tracks; (b) the specific methodology used for etching zircon grains in Teflon® mounts; and (c) etching times and conditions from zircon age standards. For counting FT densities, the questions included total magnification and objective type (oil or dry). For etching, important variables include the composition of the etchant, etching temperature, etchant vessel, and heat source. The etching times for typical standards used for zeta calibration included the Fish Canyon Tuff (FCT) (27.9 ± 0.5 Ma), Buluk Tuff (16.4 ± 0.2 Ma), Bishop Tuff (0.74 ± 0.03 Ma), Tardee Rhyolite (58.7 ± 1.1 Ma), and the Mt. Dromedary Banatite (98.7 ± 0.6 Ma) (see Wagner and Van den haute, 1992; Hurford, 1998). For a point of comparison, similar data were summarized from published papers that appeared in print between 1983 and 1999 (Table 2). Many papers are not listed in this tabulation (> 60%) because they lacked sufficient information about the details of their methodology.

3. Results

3.1. Etchant composition

Most researchers use molten KOH:NaOH for zircon etching. Although all responding labs did not indicate the "eutectic composition", it would appear that most use the general formula laid out in Gleadow et al. (1976) and subsequent publications (i.e. 11.5 g KOH mixed with 8 g NaOH). Note, however, that the KOH:NaOH etchant mixed at a ratio of 7:5 or, alternately, mixed at a ratio of 1:1, do not seem to yield appreciable difference in reported etching times (cf. Lab 17 and 6 to others in Table 1). However, it seems apparent that a KOH:NaOH:LiOH (14:6:1) etchant dramatically increases etching efficiency and allows for much lower etching temperatures given similar etching times (tens of hours at ca. 200°C).

3.2. Etching standards

Many labs appear to have a prescribed procedure for etching standards (i.e. temperature and time are predetermined). A key aspect of differences from lab to lab is illustrated in a comparison between temperature and time for each standard. In most cases, etching times were given by respondents as a range, some with an etching-time variation of ~ 10–30%. In these cases, a midpoint was taken to simplify comparison (Table 1, Fig. 1). In general, zircon etching is done between 210°C and 230°C with the largest number of labs etching between 220°C and 230°C (~ 60%). As would be expected, zircon standards with greater total α-damage (generally those which are older) require shorter etch times (see Hasebe et al., 1993). Some labs report that etching of unknowns and standards proceeds in a stepwise fashion until the sample is well etched: in these cases, the grain mount is etched for increments of time (ca. 1–10 h). After each etching increment, the Teflon® mount is removed from the oven, rinsed, and track quality is visually assessed.

3.2.1. Fish Canyon Tuff

The FCT is the most widely used age standard in the FT analysis of zircon, and for many labs it is the only age standard used. Several important aspects of etching times for the FCT are worth noting. First, if all the data are considered, there is no clear relationship between etching time and temperature. However, for etching times greater than ~ 20 h, it would appear that there is a negative correlation between temperature and etching time, as would be expected for the etching response for ceramic material: longer etching times are required at lower etching temperatures (i.e. Gleadow et al., 1976). At shorter etching times, however, the data are quite scattered, which could be due to incomplete etching or inadvertent pre-selection of high-uranium, well-etched grains (discussed below). Two labs with dramatically different etching times supplied counting data (FCT) which are introduced and discussed in the "Interpretation" section below (denoted Lab #1 and Lab #2 in Table 1, Figs. 1 and 2).

3.2.2. Other standards

Data for other standards are sparse, and it seems clear that the FCT is the only routinely used age standard in the FT analysis of zircon. The few data that do exist give a limited view of the etching characteristics of other age standards. Several observations of the Buluk Tuff Member of the Bakata Formation suggest a weak negative correlation between etching time and etching temperature (similar to the FCT above). These data do suggest that there is a fairly predictable etching response (see Fig. 1).

Table 1
Etching conditions and counting parameters for zircon in different FT labs

Lab	Mag.	Obj.	Etchant	Heat source	Vessel	Temp.	FCT	BLK	TR	BT	MtD
Labs using microscopes with dry objectives											
02	1250×	Dry	KOH:NaOH	Hotplate	Teflon	228	26[a]	30[a]	—	—	—
03	1600×	Dry	KOH:NaOH:LiOH[b]	Hotplate	Ceramic	207[a]	te	—	—	—	—
05a	1500×	Dry	KOH:NaOH	Oven	Teflon	240[a,c]	33[a]	43[a]	—	—	—
05b[d]	3000×	Dry	KOH:NaOH	Oven	Teflon	227	—	—	—	—	—
06	1250×	Dry	KOH:NaOH[e]	bath	Teflon	225	23	—	—	—	—
07	1600×	Dry	KOH:NaOH	Oven	Teflon	221	47	—	—	—	—
08	1250×	Dry	KOH:NaOH	Oven	Teflon	210	13[a]	—	—	—	—
13	1562×	Dry	KOH:NaOH	Hotplate	Teflon	210	60	—	—	—	—
14a	2000×	Dry	KOH:NaOH:LiOH	Oven	Teflon	200	40	—	—	—	—
14b[d]	2000×	Dry	KOH:NaOH	Oven	Teflon	220	—	—	—	—	—
15	1250×	Dry	KOH:NaOH	Oven	Teflon	230	30	—	—	—	—
17	1000×	Dry	KOH:NaOH[e]	Hotplate[f]	Teflon	225	25	50	22	—	8
18	1600×	Dry	KOH:NaOH	Ov/hp	Platinum	210[a,g]	—	—	—	—	—
Labs using microscopes with oil objectives											
04	1000×	Oil	KOH:NaOH[h]	Ov/hp	Teflon	233[a]	21	—	—	—	—
09	1000×	Oil	KOH:NaOH	Oven	Teflon	205	—	—	—	—	—
10	1600×	Oil	KOH:NaOH	Hotplate	Teflon	225	28[a]	39[a]	28[a]	46[a]	24[a]
11	1250×	Oil	KOH:NaOH	Oven	Teflon	214	53[a]	55[a]	40[a]	—	—
12	1500×	Oil	KOH:NaOH	Oven	Teflon	230	22	—	—	—	—
01	1250×	Oil	KOH:NaOH	Oven	Teflon	220	4	—	—	—	—
16	2000×	Oil	KOH:NaOH	Hotplate	Teflon	220	—	—	—	—	—
18	1563×	Oil	KOH:NaOH	Oven	Platinum	230	30	—	—	—	—
19	1250×	Oil	KOH:NaOH	Hotplate	Platinum	215	11	13	—	10	5

Note: te, till etched; FCT, Fish Canyon Tuff; BLK, Buluk Tuff; TR, Tardee Rhyolite; BT, Bishop Tuff; MtD, Mt. Dromedary Banatite. "Mag." refers to total magnification including objective, tube factor, and eyepiece. "Obj." refers to the type of objective used. "Temp." is in °C. Routinely, the etchant is placed in a container (Teflon®, ceramic, or platinum) and that container is held at a constant temperature by some heat source.

[a] Midpoint of range shown.

[b] Etchant ratio of 14:6:1.

[c] Low temperature (225°C for high track density samples, but higher temperatures (∼ 250°C) for samples with a low track density).

[d] Alternate methodology used.

[e] Etchant ratio of 1:1.

[f] Modified to reduce heat transfer from Teflon dish.

[g] High track density samples etched at 205°C, others at 210–215°C.

[h] Etchant ratio of 10:7.2 (all others not stated).

3.3. Special etching techniques

Several labs report using alternate methodologies for etching either: (a) old high-density zircon ($> 10^7$ tracks/cm^2); or (b) young samples with little radiation damage and few FTs. For old grains, these techniques include giving the samples a shorter etching time or reducing the etching temperature. Alternately, high densities on zircons etched by a standard methodology are counted at a higher magnification (∼ 1600–3000×). Young samples are etched at higher temperatures (> 230°C, as high as 250°C) or are etched for longer times (generally ca. 40–100 h).

4. Interpretation

The results from the FCT are amenable to analysis because it is so widely used, it has a known cooling age. A striking finding is that reported etching times for the FCT vary between 4 and 60 h. In part, this wide variation reflects an acceptable variation in etching response for zircon with α-damage, and it is reassuring to see that the system has a wide response. In detail, however, it would be even more reassuring if the conditions were more consistent between labs to facilitate interlaboratory comparison.

There are several possible explanations for the observed variation in reported etch times for the FCT: (i) different labs count under-etched, well-etched, or over-etched standards; (ii) some unrecognized variable affects etching response between different labs; (iii) natural variation in the uranium content of zircon grains of the FCT allow for a wide range of etching conditions; and (iv) temperature plays a crucial role in the etching response. To address the first explanation, a comparison of mean track size (or etch pit size) would have to be made between labs. As such, this idea is difficult to

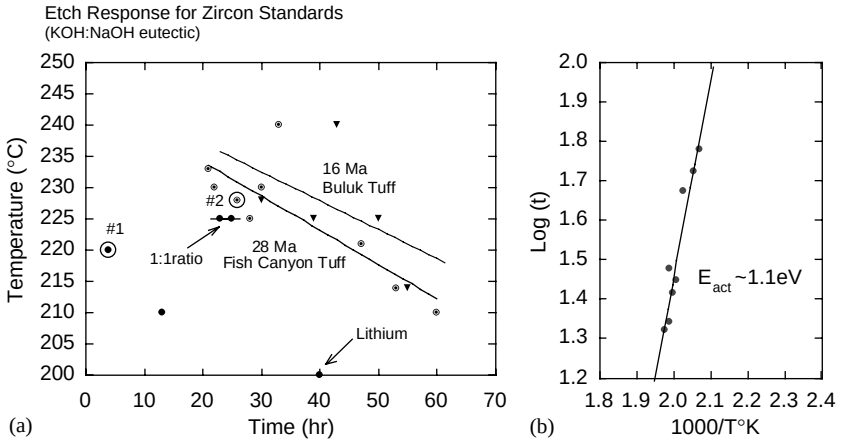

Fig. 1. (a) Relationship between etching time and temperature for the etching of standards (see Table 1 for data). Additional data from Laboratories #1 and #2 (labeled) appear in Fig. 2. Symbols: Square—Buluk Tuff; circles—Fish Canyon Tuff; circle in circle—FCT data used for best-fit regression (also for (b)); circle with bar—KOH:NaOH mixed at a ratio of 1:1. (b) Arrhenius relationship of etch times for the FCT (only solid circles in (a) are plotted here).

Table 2
Papers with zircon procedures from in different FT labs (1984–1999)

Paper	Mag.	Obj.	Etchant composition	Etch temp.	Etch time	Total FT age range	Publication date
1	1250×	Oil	KOH:NaOH	210	36–72	79–97	1989
2	1250×	Dry	KOH:NaOH	205	4–40	3–50	1998
3	1000×	Dry	KOH:NaOH	225	20–30	40–500	1996
4	1250×	Oil	KOH:NaOH	225	5–10	20–80	1988
5	1250×	Oil[a]	KOH:NaOH	230	4–30	60–300	1986
6	1500×	Dry	KOH:NaOH	220	18	20–30	1986
7	1250×	Oil	KOH:NaOH	220	6–12	25–250	1994
8	0925×	Dry	KOH:NaOH	225	10–30[b]	50–200	1993
9	1250×	Oil[a]	KOH:NaOH	220	10–15	20–30	1991
10	1250×	Oil	KOH:NaOH	225	15–25	20–180	1994
11	1652×	Dry	KOH:NaOH	228	10–60	10–1000	1999
12	1563×	Oil	KOH:NaOH	230	5–10	100–600	1984
13	1250×	Oil[a]	KOH:NaOH	230	8–20	4–30	1986
14	1250×	Oil[a]	KOH:NaOH	225	6–10	50–200	1994

Note: Representative sample of papers only, many others exist. Note, however, that this list only includes those papers that give enough information to fully reconstruct methodology: many were rejected from consideration because that lacked enough information. "Mag." refers to total magnification including objective, tube factor, and eyepiece. "Obj." refers to the type of objective used (oil immersion lens or dry). "Temp." is the etching temperature in °C. "Time" is the reported etch time for all samples (many samples lumped). "FT age range" is the total range of reported FT ages in the paper, "Publication date" is the date of publication.

[a]Assumed optical configuration.

[b]Multi-etch technique where samples were etched and counted repeatedly.

assess because track lengths or etch pit diameters are routinely measured in zircon. One possible outcome of counting under-etched grains would be low ρ_s (spontaneous track density) and a resulting low ζ value: for unknown samples, under-etching results in calculated grain ages that are too young.

The second explanation would be that some unrecognized variable affects the etching process. There are a number of lab-specific variations that might affect etching: (a) The re-use or repeated use of etchant, which might progressively alter its composition and reduce effectiveness. It seems likely from the author's experience that re-used etchant, especially after significant loss of grains from a previous etch, is not as effective as a new, unused batch. Therefore, re-use of etchant would increase the etching time needed for full track revelation. (b) The heating and cooling of a

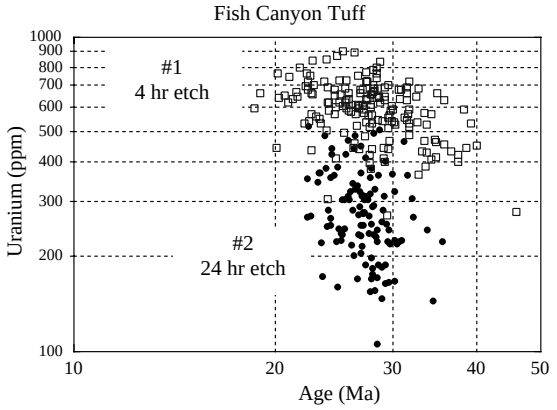

Fig. 2. Relationship between uranium content and calculated age for the ~ 28 Ma FCT age standard at Lab #1 and Lab #2 which use dramatically different etching times. Variation in ages about the accepted 28 Ma age of the sample is normal statistical variation. Symbols: squares—Lab #1, etched for 4 h; circles—Lab #2, etched for 24 h.

sample that is repeatedly removed from the etchant. Two labs indicate that etching time increases if the samples are removed from the etchant repeatedly to assess track quality, so it seems that etching is more rapid if it is done without interruption (see Hasebe et al., 1993). (c) Contaminants in the etchant may significantly affect etching. This effect may be especially important in the case of sulfides (esp. pyrite) that might be introduced to the etchant after having been mounted in the Teflon® along with the zircons (Kowallis et al., 1996). To avoid possible contamination, some labs hand pick zircon grains, remove pyrite with aqua regia (aka nitrohydrochloric acid), or use a short pre-etch to dissolve impurities early in the etching cycle. It is also possible that tweezers used to remove the mounts from the molten etchant may introduce trace metals such as iron.

The third explanation is that all etching times (long, intermediate, or short) are well suited to a subset of the overall population of grains. Because all the grains in the FCT cooled at the same time, this possibility is dependent on the natural grain-to-grain variation in α-damage, which is mainly a function of uranium content (Th^{232} has only a minor affect in α production in an average zircon: Garver and Kamp, 2002). Therefore, short etches would reveal tracks only in high-uranium grains, and long etches would reveal fission tracks in low-uranium grains with many over-etched grains.

To address the issue of whether etch time captures a uranium-dependent subset of the population, data were requested from two labs with very different standard etch times for the FCT (see Fig. 2, Table 3). Note that analysts from both labs have ζ values within about 10% of each other which suggests that the result is well calibrated. As would be predicted if both analysts counted properly etched tracks, the short etch reveals well-etched tracks in high-uranium grains and the longer etch captures low-uranium grains (Fig. 2). Because most labs appear to etch the FCT for 20 + h, one could infer that the population of grain ages counted by the FT community has a mean (more likely a median) of about 300 ppm. I am not aware of any unbiased summary of uranium content in the FCT, but I would suggest that 300 ppm is probably near the median, because when etching mounts, most analysts probably try to etch for the maximum number of countable grains (see Garver et al., 1999 for a judicially biased summary). Schmitz and Bowring (2001) report U/Pb data with a mean of 467 ppm for a number of zircon analyses (ca. 31, but some are multiple grains), but even these data are biased because fractions were hand picked prior to analysis. For FT analysis, if most grains are ~ 300 ppm, then Lab #1 captures only a small fraction of the total datable population.

An interesting outcome of such a comparison is that this inadvertent pre-selection of high-uranium grains at Lab #1 results in grains with about twice the single-grain precision (because of higher track density), and therefore, a higher theoretical precision in calculated ζ values. In this case, single-grain precision is about twice that of the longer etch and average uranium content is about twice as high in the short etch. However, the higher track density may make counting more difficult due to significant track overlap. Another implication of this finding is that it might not really matter how long one etches the FCT as long as the grain mount has enough grains in which tracks will be developed by the target etching time. Nonetheless, if the preceding scenario is correct, the majority of grains would be developed at temperatures between 220°C and 230°C with etching times of 20–30 h (for 210–220°C the etching time would be 40–65 h). It seems clear that a better understanding of the etching response of the FCT is needed. Likewise, studies aimed at quantifying the spectrum of U content in the FCT, and other standards, would be very useful.

The fourth explanation is that temperature fundamentally affects the etching process and that variation in true effective temperature relative to the reported observed temperature is caused by: (a) repeated removal of the mount from the etchant, and (b) improper heat source calibration (i.e. variation between set temperature and actual temperature of the etchant in either an oven or on a hotplate). The etching response for the FCT at etching times > 20 h suggests that as in most minerals, etching rate increases with temperature. The data from this survey suggest that a 10°C variation in temperature (between 230°C and 220°C) results in a difference in etching time of about a factor of two (see best-fit line in Fig. 1). Therefore, subtle variation in a lab setup may result in important differences in reported etching temperatures. It seems clear that further experiments aimed at fully defining the etching response of the FCT under different temperature conditions are needed.

5. Discussion

At the outset of this paper, it was noted that with more uniformity between labs, the FT community may be able to measure consistent, meaningful data from zircon. Is this in fact a problem? In practice, we are not sure. The results of this survey might be interpreted as showing that there are many ways to successfully etch zircon. However, these data may also indicate that etching is problematic and therefore ages are suspect. Anecdotal evidence suggests that a number of labs do have trouble in etching zircon, or just simply do not routinely analyze zircon due to problems associated with proper track revelation. There is little hard evidence to suggest that results are variable (or not variable) from lab to lab. In the past, researchers have participated in a voluntary inter-laboratory comparison of well-characterized samples (associated with the International Fission Track Conference, which meets every 4 years). The last survey was done in 1992 (Miller et al., 1995), but zircon has not been included in this comparison since 1984 (Miller et al., 1985), and the FT community has not had an inter-laboratory comparison since. In the1984 study, 18–20 analysts reported results for two samples of known age, one of which was the FCT. For this sample, nearly 30% of the reported ages were outside 2σ of the known age. Of these, 80% were ages that were too young (possible reflecting under-etched grains, or poor track recognition). The authors note that these errors may be due to problems with: (1) sample preparation, (2) etching, (3) microscope setup including counting and track identification, and (4) other calibration issues (Miller et al., 1985).

Despite careful attention to etch time and etch conditions, a typical mount will have grains that are under-etched, well-etched, and over-etched. Therefore, a crucial aspect of the analysis of zircon is that the analyst selects properly etched grains to count, and that all tracks are properly identified. While this aspect of counting can be difficult to monitor, it is likely that practice and experience results in consistent and meaningful data. This consistency is reflected in the fact that zeta values are similar (usually within 10%) from lab to lab and individual zeta values for a single analyst become more uniform with time (Hurford, 1998). Most analysts use a uniform criteria for selection of countable grains, which include counting only grains with: (1) surfaces parallel to the crystallographic *c*-axis; (2) well-defined polishing scratches and tracks indicating a low bulk etch rate; (3) well-etched tracks parallel to *c*-axis in grains with low track densities (see below) (Gleadow, 1981; Hurford and Green, 1983).

Grains with very low accumulated α-damage have an anisotropic etch response, which means that the bulk etching rate is different in different crystallographic orientations. The slowest rate is parallel to the *c*-axis, so one needs to ensure that etching proceeds long enough to reveal tracks in this orientation. For zircon, this problem is especially acute for grains with very low track densities and correspondingly low α-damage (grains with typical uranium concentrations less than 5 Ma or so have this problem). In practice, this means that tracks parallel to *c*-axis take two or three times as long to etch as compared to tracks perpendicular to *c*-axis (see discussion in Gleadow, 1981). So, for those with anisotropic etching, it is important to etch long enough to reveal tracks parallel to *c*-axis. However, in very young grains with few or no tracks (typically only several million years old), this criteria becomes hard to follow and one needs to rely on a long etch and an assessment of polishing scratches. Greater and greater α-damage leads to increasing isotropy in zircon, and the bulk etching rate is more or less uniform in all directions.

6. Conclusions

Most analysts experienced with FT dating zircon would agree that there are a number of factors that determine if accurate ages are being produced from lab to lab. One factor may be etch time, and another, perhaps more important, is the proper recognition of well-etched grains by the analyst. Despite the routine use of zircon for FT dating, standardization for etch time and etch conditions are lacking, certainly in comparison to procedures for apatite. Variation in α-damage and therefore the solubility of zircon causes variations in etching times from sample to sample. As long as individual labs are well calibrated, there may not be a need for widespread adoption of etching conditions for certain standards. However, because etching time, temperature, and etchant composition are so fundamental to the etching response of both standards and unknowns, it is critical that these procedural data are clearly reported in publications.

As it seems clear that the FCT and the Buluk Tuff are the primary means of calibration for the zircon FT system, a standardized etching procedure would be useful, especially, as a number of new emerging labs tackle this issue. The following recommendation is based on the preceding discussion, the principle methodology of operating labs, and the practicality of setting up new facilities: (1) Two age standards should be used for age calibration, with the Fish Canyon Tuff and the Buluk Tuff as the primary candidates (i.e. see Hurford, 1998). (2) Clean, sulfide-free zircon separates should be embedded in PFA or FEP Teflon® and polished using standard procedures. (3) Etching should be done in covered Teflon® dishes in well-calibrated thermostatically controlled oven at 228–230°C. (4) Under these conditions, the FCT should be etched for 28–30 h, and the Buluk Tuff should be etched for 30–34 h in new, unused etchant. (5) Fission tracks should be counted between 1000× and 1600×, but the exact setup of objectives and the microscope seem to make little difference.

It seems clear that more work is needed to better address the etching response of zircon. The preceding discussion involves grains with a rather narrow range of α-damage, and clearly the objective is to date samples of unknown ages. For most studies, samples with cooling ages less than 1 Ma

and up to 1 Ga have etch times that vary between 2 and 100 h, depending almost exclusively on inferred α-damage. Most researchers rely on experience to guide them through the etching procedure for unknown samples, but a more quantitative assessment is needed to facilitate wider use of this technique.

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