DATA REPOSITORY

Supplemental File S1: detrital zircon U-Pb age data including Concordia ages, zircon and sample descriptions and sample locations

Supplemental File S2: apatite fission track methodology

Supplemental File S3: apatite fission track sample location, age and length data, and electron microprobe analyses

Supplemental File S4

SUPPLEMENTAL FILE S2: APATITE FISSION-TRACK LABORATORY PROCEDURES

Apatite separates for all samples were obtained from crushed and separated material using standard gravimetric and magnetic mineral separation techniques. Apatite grain mounts were prepared by Paul O’Sullivan at the GeoSep Services (GSS) facilities in Moscow, Idaho. Spontaneous track counts and confined track length measurements were performed by Paul O’Sullivan using nonpolarized light at 2000x magnification. Laser ablation–inductively coupled plasma–mass spectrometer (LA-ICP-MS) analyses of samples used in age determinations were performed using the Element2 mass spectrometer located at the Washington State University School of Earth and Environmental Sciences GeoAnalytical Laboratory in Pullman, Washington.

A general discussion of the methods undertaken to process and analyze samples by GSS is presented below; see Donelick et al. (2005) for a complete and detailed discussion of these methods and their justification. For each sample subjected to apatite fission-track analysis (AFT), at least one 1cm² grain mount, consisting of apatite grains immersed in epoxy resin, was prepared, cured at 90 °C for 1 h, and polished to expose internal surfaces of the apatite grains. After polishing, mounts were immersed in 5.5N HNO₃ for 20.0 seconds (±0.5 seconds) at 21 °C (±1 °C) to reveal all natural fission tracks that intersected the polished grain surfaces.

The feasibility of measurement of apatite fission-track grain ages and track lengths was assessed by scanning the polished and etched grain mount to determine if any dateable apatite grains were present. Measurement of fission-track parameters was considered feasible if more than one dateable grain was observed.

Representative kinetic parameters ($D_{par}$—the maximum diameter of fission track etch pits at their intersection with the polished and etched, c-axis-parallel apatite surface, which is used as a proxy for the solubility of fission tracks in their host apatite grains) were measured and spontaneous (natural) fission-track densities were counted for each grain considered suitable for dating. Between one and four etch pit diameters were measured and an arithmetic mean $D_{par}$ value was calculated for each datable grain.
LA-ICP-MS Analysis

Grains were then revisited using the LA-ICP-MS to make spot analyses to determine U, Th, and Sm concentrations of each grain for which natural fission-track densities had been previously determined. A single stationary spot of 16 µm diameter was used for each grain, centered in the approximate center of the area where tracks had been counted. The depth of each pit is listed in Table S1. Note that if optical examination suggested that natural track densities were even moderately inconsistent within a grain, which is evidence of U zoning, that grain was not dated.

For apatite, the fundamental assumption is made that Ca occurs in stoichiometric amounts in all grains analyzed. The isotope $^{43}$Ca is used as the indicator of the volume of apatite ablated. Samples were ablated in a helium atmosphere to reduce condensation and elemental fractionation. A total of 30 scans for $^{238}$U, $^{232}$Th, $^{147}$Sm, and $^{43}$Ca were performed for each spot analyzed. Of these scans, ~10 were performed while the laser was warming up and blocked from contacting the grain surface, during which time background counts were collected. Once the laser was permitted to hit the grain surface, a cylindrical pit was excavated to a depth beyond which uranium did not contribute fission tracks to the etched grain surface. Between 15 and 20 scans performed during pit excavation were required to reach this depth. The depths of a representative number of laser pits were measured and the $^{238}$U/$^{43}$Ca value for each pit as a whole was determined based on the weighted mean of the $^{238}$U/$^{43}$Ca value for individual scans relative to the depths from which the ablated material was derived (see Hasebe et al. 2004; Donelick et al. 2005).

Fission-Track Age Measurement

Fission-track ages and errors were calculated using: (a) the ratio of the density of natural fission tracks present in the grain to the amount of $^{238}$U present and (b) a modified version of the radioactive decay equation that includes a LA-ICP-MS zeta calibration factor (see Equations 1b for age equation and 2b for error calculation in Donelick et al. 2005). The zeta calibration factor is determined for each sample analyzed during each LA-ICP-MS session by analyzing the U:Ca ratio of apatite calibration standards with known ages at the beginning and end of each LA-ICP-MS session. The standard used are Durango apatite, 30.6 ± 0.3 Ma.

Calculation of a single pooled AFT age for each sample takes into account the distribution of all of the individual grain ages and their uncertainties, which are a function of the number of spontaneous tracks counted over a known area, the U content determined by LA-ICP-MS, and thermal history. Only pooled ages are reported as these incorporate original track counts and isotopic values for each grain, and therefore are most representative of the original data generated for each sample, even when multiple grain-age populations might be present as suggested by the Chi2 value.
Apatite Fission-Track Length Measurement

In order to enhance the number of confined tracks available for length measurement (e.g., Donelick and Miller 1991; Donelick et al. 2005), subsequent to fission-track age determination the grain mounts were irradiated with ~10^7 tracks/cm² fission fragments from a ^{252}\text{Cf} source in a vacuum chamber. Donelick and Miller (1991) demonstrated that irradiating apatite grains with ^{252}\text{Cf}-derived fission fragments could yield a 20-fold increase in the number of available fission tracks for length measurement. The ^{252}\text{Cf}-irradiated apatite mounts were re-etched using the same formula as before in order to reveal horizontal, confined fission tracks within the apatite grains. Only natural, horizontal, confined fission tracks in apatite with clearly visible ends were considered candidates for length measurement. The length and crystallographic orientation of each fission track were determined using a digitizing tablet interfaced with a personal computer. The precision of each track length is estimated to be ± 0.20 μm; the precision of each track angle to the crystallographic c-axis is estimated to be ± 2 degrees.

REFERENCES CITED


Field sample number: 008MBB307A-1
Pooled AFT age = 58.2 +/- 3.4 Ma
χ² probability= 25%
a) measured track length histogram and model track length distributions calculated using AFTINV (Issler et al. 2005).
b, c) single grain AFT ages vs. eff Cl and Dpar; note the lack of a systematic change in age with these variables
d, e) AFT length vs eff Cl and Dpar
f) RadialPlotter plot showing estimated component populations (peaks) of the fission-track grain-age distribution for this sample, calculated using RadialPlotter program of Vermeesch (2009).
g) histogram of vitrinite reflectance %Ro calculated for the model cooling curves using the kinetic model of Nielsen et al. (2017). Despite allowing the %Ro to vary between 0.05 and 0.07, only %Ro values between 0.68 and 0.7 were permitted by the data.