

SUPPLEMENTARY FILE S1

BK FISSION TRACK ANALYTICAL METHODS

For samples analysed by fission track by BK at the University of Melbourne, polished apatite grains were etched in 5M HNO₃ for 20 seconds at 20°C to reveal the fossil tracks. A gold coating with a 5-7 nm thickness was applied to the etched mounts under vacuum so as to enhance the reflectivity of their polished surfaces, and minimize internal reflections within grains and from shallow-dipping tracks, thereby considerably improving the quality of reflected light images. Apatite grains with polished surfaces parallel to the crystallographic c-axis and relatively homogeneous track distributions, with minimal dislocations and other surface imperfections, were selected for analysis.

Recent developments in digital microscopy, image analysis and computer software (designed for capturing high resolution images) have provided opportunities for a new automated counting approach for apatite fission-track analysis software (Gleadon et al., 2009). Hence, for fission track data presented in this report the software (TrackWorks© and FastTracks©) recently developed by the Thermochronology Group at the University of Melbourne and Autoscan Systems was used. This software suite controls a Zeiss M1m digital microscope fitted with a high-resolution camera and an AutoScan© stage and at a total magnification of x1000 under both transmitted and reflected light is employed for capturing a z-stack of images of the apatite crystals. The images can be used as a virtual microscope enabling the operator to examine them by switching between reflected and transmitted light and also by scrolling through the captured z-stack to check track morphology. This approach also improves accuracy by optimizing the area over which spontaneous tracks are counted to match the grain shape, as well as the visualisation of crystals on a computer monitor. Further, a computer binary threshold routine performed on the digital images allows the software to discriminate between tracks from non-track features (e.g. dust, inclusions, polishing scratches), with similar or better precision than a human operator.

A further benefit of the above procedure is that a permanent digital record of data from analysed crystals is stored and available for later inspection, even after grains have been partly destroyed by laser ablation at a later stage. As part of the procedure, coordinates for each grain used for capturing fission track data are also stored for later export for laser ablation ICP-MS analysis.

Fully etched confined track lengths and etch pit diameters (Dpar) of grains were also measured from digital images after the c-axis had been determined using FastTracks©. Digital magnification that can be generated from the high-resolution images in the measuring tools in this software enables the user to determine confined fission track lengths and Dpars with improved accuracy. In addition, several Dpar measurements were carried out on each grain on which fission track counts and length measurements were made. Magnification used for length measurements was identical to that for track counting and the angle of track lengths in relation the c-axis was also measured. The uranium content of grains on which spontaneous track counts had been counted was determined using an Agilent 7700x Series ICP-MS (Inductively Coupled Plasma Mass Spectrometry) system coupled with a pulsed (Q-switched) New Wave Nd:YAG laser with a wavelength of 213 nm. For standardization NIST-612 glass and an in-house apatite were used. The method essentially follows the protocol described by Hasebe et al., (2004). Any grains that showed obvious visual fission-track inhomogeneity and had clear U variations were discarded from age calculations to prevent erroneous age estimates due to parent isotope zonation.