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Analyst and etching protocol effects on the reproducibility of apatite confined fission-track length measurement, and ambient-temperature annealing at decadal timescales

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Abstract

Previous inter-laboratory experiments on confined fission-track length measurements in apatite have consistently reported variation substantially in excess of statistical expectation. There are two primary causes for this variation: (1) differences in laboratory procedures and instrumentation, and (2) personal differences in perception and assessment between analysts. In this study, we narrow these elements down to two categories, etching procedure and analyst bias. We assembled a set of eight samples with induced tracks from four apatite varieties, initially irradiated between 2 and 43 years prior to etching. Two mounts were made containing aliquots of each sample to ensure identical etching conditions for all apatites on a mount. We employed two widely used etching protocols, 5.0 M HNO_3 at 20 °C for 20 s and 5.5 M HNO_3 at 21 °C for 20 s. Sets of track images were then captured by an automated system and exchanged between two analysts, so that measurements could be carried out on the same tracks and etch figures, in the same image data, allowing us to isolate and examine the effects of analyst bias. An additional 5 s of etching was then used to evaluate etching behavior at track tips. In total, 8391 confined fission-track length measurements were performed; along with 1480 etch figure length measurements. When the analysts evaluated each other's track selections within the same images for suitability for measurement, the average rejection rate was ~14%. For tracks judged as suitable by both analysts, measurements of 2D and 3D length, dip, and *c*-axis angle were in excellent agreement, with slightly less dispersion when using the 5.5 M etch. Lengths were shorter in the 5.0 M etched mount than the 5.5 M etched one, which we interpret to be caused by more prevalent under-etching in the former, at least for some apatite compositions. After an additional 5 s of etching, 5.0 M tracks saw greater lengthening and more reduction in dispersion than 5.5 M tracks, additional evidence that they were more likely to be under-etched after the initial etching step. Systematic differences between analysts were minimal, with the main exception being likelihood of observing tracks near perpendicular to the crystallographic *c* axis, which may reflect different use of transmitted vs. reflected light when scanning for tracks. Etch figure measurements were more consistent between analysts for the 5.5 M etch, though one apatite variety showed high dispersion for both. Within a given etching protocol, each sample reflected a decrease of mean track length with time since irradiation, giving evidence of 0.2–0.3 μm of annealing over year to decade timescales.

Keywords: Fission track, etching procedure, step etching, ambient temperature, annealing, confined track length