Etching of fission tracks in monazite: Further evidence from optical and focused ion beam scanning electron microscopy

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ABSTRACT

A series of experiments on monazites from Victoria, Australia, is presented to further understand their fission track etching properties. Using a 6 M HCl etchant at 90 °C, SEM images on crystal (100) pinacoid faces reveal well-etched rhombic spontaneous fission track openings. Average rhombic etch pit diameters $D_{pc}$ and $D_{pb}$, parallel to the crystallographic $c$- and $b$-axes are 0.81 ± 0.20 $\mu$m and 0.73 ± 0.26 $\mu$m, respectively. An angular distribution experiment on (100) faces found that spontaneous fission tracks initially etch anisotropically, being preferentially revealed at an azimuth of 90° to the crystallographic $c$-axis up to ~60 min of etching. As etching continues, however, the distribution becomes progressively more uniform and is essentially isotropic by 90 min. Two experimental methods determined the rate at which the etchant penetrated along the lengths of implanted $^{252}$Cf fission tracks. This involved the application of a focused ion beam scanning electron microscope (FIB-SEM) to mill progressively into slightly etched monazite crystals followed by an etch-anneal-etch approach. Results indicate that at least the greater part of the etchable ranges of the latent fission tracks were penetrated by the 6 M HCl etchant within the first few minutes. Continued etching to 5 min indicates that track etching slows down toward the ends of the tracks, but the maximum ranges are estimated to be reached after 5–15 min, which represents the longest time the latent segments of the tracks are exposed to potential annealing at the etchant temperature. Taking into account that implanted $^{252}$Cf fission tracks in monazite anneal on average ~4% of their length at 90 °C after 1 h (Jones et al. 2019), suggests that a much shorter duration for exposure to this temperature causes less than ~1% of fission track length reduction during etching.

Keywords: Monazite, fission tracks, etching, FIB-SEM, etch-anneal-etch

INTRODUCTION

A series of experiments presented by Jones et al. (2019) led to a first-order understanding of monazite fission track etching properties. An alternative 6 M HCl [1:1 HCl (37%): H$_2$O by volume] etchant at 90 °C for 60–90 min was introduced, rather than using concentrated 37% HCl (12 M) at 90 °C for 45 min [originally proposed by Shukoljukov and Komarov (1970)]. The weaker etchant proved to reduce both grain corrosion and grain loss from epoxy mounts while still producing well-etched fission tracks. Results from an electron backscatter diffraction (EBSD) experiment demonstrated that when using standard mounting techniques (see Kohn et al. 2019 for procedure), the majority of euhedral grains would settle on their (100) pinacoid faces as the dominant orientation. A 90 °C isothermal annealing experiment illustrated the degree of track length reduction of implanted $^{252}$Cf fission tracks under laboratory timescales. This experiment also demonstrated the low-temperature sensitivity of fission tracks in monazite, with a total of ~4% annealing occurring after 1 hour and a maximum of ~20% reduction after 15 h. The final experiment of the study indicated that there is some compositional control (principally by U and Th) on track etching rates between different monazite grains, suggesting that there is a weak relationship with accumulated radiation damage.

These experiments were an important first step toward understanding the etching characteristics of fission tracks in monazite. However, further information is required to establish a standardized protocol for track etching and measurement. It is evident from earlier studies that tracks in monazite undergo substantial annealing at relatively low temperatures (e.g., Weise et al. 2009), thus a key concern is the question of the degree of track annealing that occurs using the 6 M HCl etchant at 90 °C on laboratory timescales. The question also arises as to the rate of etchant penetration along the length of tracks. In one of the earliest track etching experiments, Price and Walker (1962) demonstrated that etchants penetrate almost instantaneously along the highly reactive core of latent tracks in mica. Observations made by transmitted electron microscopy showed that well-defined hollow channels in various micas appeared in <1 s. Recent experiments in apatite also found that in the earliest stages, the etching rates of both latent spontaneous and induced fission tracks in apatite are significantly higher at their cores (Tamer and Ketcham 2020). A similar relationship might be assumed to occur in other minerals, including monazite, meaning that the actual exposure of annealable latent tracks to 90 °C before they are etched is most likely negligible, and certainly much less than the ~4% shortening that would result if the latent tracks had been