Manuscript gchron-2021-28 - Revision 1

Short communication concerning experimental factors affecting fission-track counts in apatite

Carolin Aslanian, Raymond Jonckheere, Bastian Wauschkuhn, and Lothar Ratschbacher

Replies to the reviewers' comments

Overview of substantial corrections

As explained in our detailed replies to reviewers *Drs. M. Tamer* and *H. Iwano*, we accepted almost all their comments. The few exceptions concern matters of phrasing and one Figure. Instead, we added two Figures, replaced Figure 4, and provided three more in supplement. Our main corrections are:

1. We added an image (Figure 3) of a basal section illustrating the loss and gain of tracks from different causes.

2. We corrected and added some detail to Figure 3 (becomes Figure 4). This forms the basis for a comparison of the measurement data with numerical predictions based on the etch model of Aslanian et al. (2020). The model predictions are explained in Figure 5, which replaces our previous Figure 4. The new Figure illustrates the same principle as the previous in greater quantitative detail.

3. We added significant details to Figure 6 (becomes Figure 7), which enable us to address the matter of transmitted-light vs. reflected-light counts in a more detailed and convincing fashion (see replies to Dr. *H. Iwano's* comment to Figure 5). We added Figure 8, illustrating the extended discussion.

4. We added the missing data on the sizes of the etched-track openings to the data supplement.

5. We provide reflected- and transmitted-light images illustrating the loss and gain of tracks with etch time in supplement.

The reviews of *Drs. M. Tamer* and *H. Iwano* offered us an opportunity to take a fresh look at our data. This permitted us to pull together further strands of numerical evidence, and to present a fuller interpretation and a much stronger case for our conclusions than before. We expect that the complete corrections will lengthen our text by 50%, and therefore ask the editor to consider the suggestion of one reviewer to treat our manuscript as a full article instead of a short communication.

Freiberg, 16 November 2021.

C. Aslanian
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Reviewer #2 (Dr. H. Iwano)	Replies
This paper reported experiments concerning the effects of grain orientation, polishing, etching and observation on fission-track counts in apatite. The paper also showed results were systematically obtained and theoretically explained in the framework of a recent etch model. I think this is an article worth reading for FT researchers, therefore my recommendation would be publish this paper as a short communication. The following comments should be further developed for publication.	We are grateful to the reviewer for supporting the publication of our manuscript. We have acted on all comments, as discussed below, and expanded the discussion of our etch model and track counts.
Figure 1. Explanation for regression line is needed in the caption.	Corrected; the caption to Fig. 1 now refers to the regression lines, and to their slopes and intercepts in Table 2.
Table 1. The track densities of B00 and B60 are clearly smaller than those of P00. Is this a difference due to U concentration? If not, which one is closer to the true track density?	Indeed; the sections were cut from different crystals. The higher track counts for P00 are due to its higher U-content; the differences between the samples are well within the range of uranium variation in Durango apatite and immaterial to the dependence of the track counts on etch time.
Figure 5. I am amazed at the number of track-shaped pits in the reflected image. Of course the authors counted them as a fission track. What are the criteria for track identification? Please describe them for each (TL and RL). If there are several etch pits detected by apatite that is totally annealed at 450 ° C, I think it can be set as the minimum noise to identify (count) fission tracks. Additional images of totally annealed samples are needed and helpful.	Corrected; we clarified our track counting criteria. In both transmitted light (TL) and reflected light (RL), we counted each distinct feature as a track that was not identifiable as a polishing feature or another defect; we do not recall observing the latter in these samples.
	We guess that the reviewer suspects that the features counted in RL but not in TL are not fission tracks. This is reasonable, as such features have not been reported before. But that is not difficult to understand. First, the shallow etch pits observed in RL (Figure 5) develop in the slowest-etching faces (Jonckheere et al., 2019; Jonckheere et al., in press). The principle illustrated in our Figure 2 explains this. Some prism faces etch >15 % faster than the slowest. Shallow etch figures are therefore less prominent or absent in faster etching prism faces. A second reason why the shallow etch pits have not been reported before is that, as far as we know, apatite surfaces are rarely polished to the standard of our sam-

ples, i.e. a nano-polish with 0.04 μm silica suspension until no scratches are visible in reflected light, even using Nomarski differential interference contrast. The scratches visible in Figure 5 (re-)appeared after etching even though they are caused by polishing. The shallow etch figures would not be distinguishable in less well polished surfaces. The third reason why the shallow etch figures have not been reported is that they cannot be counted in transmitted light, while track counts in reflected light are uncommon. Moreover someone observing them would be inclined to dismiss them, either as not being identifiable as tracks or as too difficult to count with confidence. None of this is reason to conclude that they are not tracks, however.

The reviewer proposes to etch an apatite annealed 450 °C as a decisive test. We have not done that because it would not prove what one might expect. Numerous experiments attest that at 450 °C all tracks are erased *in TL*, but TEM shows that short track segments survive (Paul, 1993; Li et al., 2010; 2011; 2012; 2014). So we can expect to see no tracks in TL, but what of RL? If we see nothing, we could conclude that the small etch figures were associated with defects with identical annealing kinetics as fission tracks, and thus likely also tracks. If we do observe shallow etch pits when we see no tracks in TL, then do they correspond to defects that are more stable than fission tracks or to short surviving fragments of tracks? Neither outcome establishes if the RL features correspond to fission tracks or not.

In our revision, we explain our reasons for interpreting them as fission tracks, or sections of tracks. The first is that we did in fact perform 450 °C annealing experiments, eleven even, with the difference that we irradiated the annealed sections to create the induced tracks. Now, if the RL-features (for short) are not fission tracks what is the likelihood that they occur in a constant proportion with the TL-tracks (for short) in all eight pre-annealed and irradiated samples (except three later re-annealed to $\rho/\rho_0 < 0.70$) with induced track densities 10-20 times higher than the fossil track densities (before partial annealing)? We further note that for those eight samples as well as the four containing fossil tracks annealed to $\rho/\rho_0 > 0.70$ there exists an almost perfect correlation ($r = 0.995$) between the TL and RL counts.

All the ρ_{TL}/ρ_{RL}-ratios before the break-up point are of the order of ~0.9, a value consistent with independent estimates of the track counting efficiencies in transmitted light (ηq_{IS}; Jonckheere and Van den haute, 2002; Enkelmann et al. 2005; Soares et al., 2013; Iwano et al., 2018).

At $\rho/\rho_0 < 0.70$, the $\rho_{TL}/\rho_{TL,0}$-ratio collapses to zero, while the $\rho_{RL}/\rho_{RL,0}$-ratio *remains constant*. The first is interpreted as a result of a break-up of the tracks into segments too short to be distinguished in TL. The second observation can be accounted for in the same way but only if the RL-features are sections of broken-up fission tracks. The last empirical fact which puts it beyond doubt that the RL features are indeed tracks or track sections is that the track counts including both the TL tracks and RL features have standard deviations which in all investigated samples are close to those of a Poisson distribution (Table 3). That could happen once by accident but not 18 times and for close to 1000 counted areas. Strong claims require strong evidence, which we believe we supplied in this case. But the

	most convincing argument of all is that all FT labs are in possession of four samples identical to ours. Everyone wishing to investigate our conclusion can go to the microscope and put it to the test.
Figure 6. Between 0.7 and 1.0, track density for RL is higher than TL. This means that the track identification criteria are different. Please describe the identification criteria for minimum track, at least.	Corrected; we explain our track counting criteria, which are straightforward, in the revised manuscript
Table 3 and Figure 6. Are there data at 450 ° C for total annealing? I am very concerned about the density of track-like defects.	Yes and no. All the samples with induced tracks had been pre-annealed at 450° before neutron irradiation. We did not put a sample apart before the neutron irradiation because the sections were intended for a different experiment. As we explained above, an annealed but un-irradiated sample would not be conclusive, and we gave compelling reasons for our interpretation of the RL features as fission tracks ($1 > \rho/\rho_0 > 0.70$) or track sections ($0.7 > \rho/\rho_0 > 0$). We question, however, that the RL features need to be a cause for great concern, as lossless fission-track counts were never possible, as the reviewer's own work on standardless FT dating shows.

Freiberg, 16 November 2021.

C. Aslanian
R. Jonckheere
B. Wauschkuhn
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