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Reply on RC2

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Author comment on "Technical note: Rapid phase identification of apatite and zircon grains for geochronology using X-ray micro-computed tomography" by Emily H. G. Cooperdock et al., Geochronology Discuss., https://doi.org/10.5194/gchron-2022-7-AC1, 2022

Response to referee comment by Anonymous Referee #2

We thank Referee #2 for their review and suggestions to improve the manuscript. We have responded to all points and made changes according to the referee's suggestions. Author responses below are given in bold.

This manuscript presents a detailed description of a microCT method for distinguishing apatite from zircon. The authors clearly lay out an optimized methodology, along with excellent figures that illustrate various aspects of the data and measurements. Besides some suggestions that I provide below for clarification, I have no issue with this manuscript being published. I appreciate the authors' efforts to develop another way to use microCT together with geochronology methods. However, I'm skeptical this method will be widely adopted for distinguishing apatite from zircon. The authors might consider shifting the manuscript emphasis in places, as suggested below, to potentially make this contribution more impactful.

In my view the problem being addressed (distinguishing apatite from zircon in mineral separates) is greatly overstated. In the vast majority of circumstances, it isn't challenging to distinguish apatite from zircon under the microscope after separation with LST. These minerals are distinct in morphology, relief, and other properties. Even for newbies, after a few hours of getting one's eyes calibrated at the microscope, it is not particularly difficult to distinguish these mineral phases. It arguably becomes more important for detrital mineral suites – it may be effective for the authors to specifically emphasize this challenge, rather than trying to argue that this is a routine problem when it really isn't and most reading this paper will know this.
Agreed that performing mineral separation with toxic chemicals is undesirable, but again this strikes me as overstated given that one can alternatively use LST and then i.d. the minerals typically without too much trouble.

We did not intend to say that distinguishing between apatite and zircon is impossible or a particularly difficult task in many sample separates. However, there can be some challenges in distinguishing small grains, grains with broken tips, and abraded detrital grains. Therefore, there is a possibility of individual grains being misidentified, which will vary based on the experience of the person picking and the sample type. In our own lab we experienced misidentified grains,

and we have heard similar stories from most other labs. We propose that microCT scanning can be an optional pre-screening or validation method which can be implemented in routine sample preparation procedures. We have clarified our phrasing and motivation throughout the manuscript, particularly in the Introduction.

Although not emphasized in the abstract or introduction, elsewhere in the paper the authors highlight attempting to distinguish apatite from titanite with microCT. These phases are even easier to differentiate than apatite and zircon, with titanite typically coming off at a more magnetic level on the frantz than apatite. It may be better to eliminate this comparison in this paper entirely.

Mineral separation techniques are not 100% effective, so any separate could potentially contain titanite (such as the separates in this study), which could be hard to distinguish from apatite in grains with poor morphology. We included titanite in the analyses to highlight some of the potential challenges with using microCT (e.g., phases with similar density), and we therefore think it is worth keeping the data in place.

It would be helpful to provide some estimate of the total time required per grain (including mineral selection, mount making, analysis, data reduction) to 1) use the proposed microCT method to distinguish different mineral phases and 2) additionally identify inclusions and acquire grain geometry information.

The total time required will depend on the level of training and familiarity with microCT data analysis as well as computer hardware. The time investment comes in the beginning as one is learning the segmentation software. Once a user is familiar with the software functions, the time investment is on the order of a couple hours maximum if they want to calculate grain-specific surface areas and volume for 3D Ft correction or grain mass estimates.

1) Distinguishing between apatite and zircon (as long as no other phases are present) can be done visually by viewing the microCT data in an appropriate software, which only takes a few minutes. Segmentation and average grayscale value computation can be done in a few steps in most microCT software packages, such as Dragonfly, and can be (semi-)automated, requiring 5-15 min to be completed. This time is per acquired volume, so it could either apply to a single layer (as reported here), or to a stack of layers (containing hundreds or thousands of grains). 2) We have identified inclusions in grains by inspecting successive grayscale slices through each grain. Identifying inclusions and classifying individual crystals this way takes <1 min per grain. Calculating grain-specific volumes and surface area are done in batch and can be (semi)-automated like the segmentation computation mentioned above in 15 min or so using Dragonfly software.

In the end, this is not significantly more time than is required to measure via 2D methods on a microscope (which does not need to be done if one uses CT), and has the added benefit of being more accurate for odd shaped grains.

We add this discussion in sections 3.5 and 3.6.

Is the microCT method for mineral i.d. faster than alternative analytical methods that could be used to identify these phases? For example, in my experience, mineral identification using an EDS system on an electron microprobe or SEM requires only a few minutes to place individual crystals on carbon tape and then seconds per grain for EDS identification. This seems faster than the microCT method described here, and EDS

systems are more common and therefore more accessible than microCT systems. If this is incorrect, then it would be helpful to clarify this in the paper.

From our experience, a single-layer grain mount can be assembled as part of the picking process, and therefore requires little additional time to prepare, which is similar to EDS. MicroCT volumes show information for all grains simultaneously. If pure phase identification is required, it can be done visually, which only takes seconds. This is faster than manually placing dozens of Raman or EDS spots. However, these types of analyses can yield information about composition, which might be preferable in some situations. Overall, microCT can be used as the main pre-screening method or it can be combined with other methods, such as the ones mentioned, for a more thorough sample characterization.

If one is going to the trouble of making the mount, then why not use the longer scan times to acquire the additional information about inclusions and grain geometry? This strikes me as a more compelling reason to use this method, and could be emphasized more strongly as a motivation in the paper. Or perhaps this could become the paper's primary motivation.

Other papers have described the procedure for grain geometry analysis and inclusion mapping (e.g., Evans et al, 2008; Cooperdock et al., 2019; Glotzbach et al., 2019). Additionally, we have a manuscript focusing on using microCT to detect inclusions in various minerals, including optimizing scan parameters and analysis procedures is in preparation. Phase identification can be one motivating factor for pre-screening apatite and zircon grains with microCT. Phase identification, as described here, can be integrated into the routine microCT data analysis for pre-screening procedures.

Lines 33-43: Suggest revising the second sentence. The characterization of the crystal shape does not matter for U-Pb and fission-track, unlike what is implied by the structure of these two opening sentences.

We have revised to sentence to be clear these are important factors for (U-Th)/He.