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Interactive
comment

Interactive comment on "Resolving the effects of 2D versus 3D grain measurements on (U-Th)/ He age data and reproducibility" *by* Emily H. G. Cooperdock et al.

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Response to: Interactive comment on "Resolving the effects of 2D versus 3D grain measurements on (U-Th)/ He age data and reproducibility" by Emily H. G. Cooperdock et al. Anonymous Referee #2 Received and published: 17 June 2019

Please find our responses below each comment. We also attached the revised manuscript and appendices with tracked changes.

General comments: The manuscript "Resolving the effects of 2D versus 3D grain measurements on (U-Th)/ He age data and reproducibility" by Cooperdock et al. provide

insight into the issue of accurately measuring apatite grain dimensions for ultimate use in (U-Th)/He age calculations. The manuscript is concise and well written with respect to identifying key problems with conventional 2D microscope approaches and adds to the literature beginning to assess 3D (CT scan) methods for better grain characterization. Please refer to the attached annotated PDF for in-depth line comments; main points are highlighted below.

I found some references to the general U-Th/He method (early development and application; Zeitler et al. '87, 90s Wolf/Farley/Ehlers/Reiners work etc.) to be lacking and additionally some recent work on age dispersion/analytical approaches for assessing age reproducibility to be uncited, and even prior 3D/CT scan work was cited later in the introduction (Evans et al.; Herman et al.; Glotzbach et al. etc.) - when statements made earlier in manuscript should cite these references as well or perhaps requires reworking of this intro. section.

Response: References have been added to the introduction, including earlier reference to previous CT work.

The authors show that when comparing 2D vs. 3D approaches for age calculations, that there is essentially no difference in the computed U-Th/He ages. While I agree that 3D-CT seemingly yields overall 'better' grain dimension(s)/morphology results, the additional time/cost put forth for CT work is not realistic for most users - i.e. the cost/benefit does not make this approach worthwhile to researchers that do not have easy CT scanner access.

Response: We do see the point of this statement, and we've made a conscious effort not to over-sell the technique (as endorsed by the reviewer in his next sentence). However, as costs continue to fall and capabilities continue to rise, and new uses are thought of and developed, we believe the utilization of this technology will continue to grow.

Page 18, Lines 20-25 best summarize this conceptually..."Overall, these data suggest

that although the 3DFT can provide a more accurate FT correction and varies from 2D estimations by âĽij2%, it has a minimal effect on the calculated sample age (1-2%) and no effect on the reproducibility for these two samples. This is not surprising, as a âĽij2% error would constitute a negligible proportion of the often cited 6% dispersion derived from analyzing age standards; error propagation indicates that removing a source of 2% error would only reduce an overall 6% error to 5.7%. This points to the importance of other factors in intra-sample dispersion, such as U-Th zonation, and/or excess He from nano-inclusions or high U-Th neighbors." This passage advocates for a more proactive approach to dealing with real and greater sources of age dispersion.

Response: We agree with this statement.

Additionally, this work utilizes apatite grains that were previously known to yield highly reproducible results (and the same holds true after their new work). This was probably the best approach to assess first-order discrepancies between 2D/3D, however, what does a sample look like that yields highly scattered age data? Does 3D help with reducing (some) dispersion? As we know, it is more common for a lot of apatite He ages to be statistically over-dispersed. I think for additional communal value the authors should characterize some other 'normal' samples.

Response: We generally agree with this statement. As the reviewer states, we purposefully selected reproducible and high quality apatite for this study as a "best case scenario" assessment. We have also now reported the results when we randomly subsample 4 grains from our dataset, to make for a more realistic test. We ran this simulation 1000 times and report the results in Figure 7. These results show more variation, and generally slightly better reproducibility in the 3D method, though still within error of the 2D, and within error of the whole population statistics. However, we do not agree that characterizing more and more-dispersed samples would be helpful; our two samples already feature differing degrees of age dispersion, and we show the FT component of dispersion is similar. Adding in more excess dispersion that we by definition don't understand will not provide further clarity.

Major Line-by-line comments (see PDF for all): Page 1, Line 12: How do you quantify eU with 2D microscope or CT information?

Response: We removed eU from this sentence to avoid confusion. eU quantification is described in Appendix A and Methods. The eU is calculated based on the U, Th and Sm concentrations using Eq. (7). The concentration data are determined by isotope dilution via the ICP-MS (see methods) and using the mass of the grain, which is calculated by the grain dimensions. Therefore, the grain measurement technique indirectly feeds into the eU concentration through the calculation of the grain mass.

Page 1, Line 16: Arguably no one uses mass for interpreting age scatter and eU (known to be underestimated)...ESR may be problematic and could be better addressed by the 3D technique. In regards to eU/ESR-age relationships being used in a meaningful way for age scatter interpretation, see recent paper by Fox et al. 2019 G-cubed "Badly behaved detrital U-Th/He ages: problems with He diffusion models or geological models?"

Response: We mention eU (which is a function of grain mass) here in reference to the effect of radiation damage on He diffusivity, which is still used for inverse modeling and age-scatter interpretation. We rephrase the sentence a bit to clarify this. We also add a reference to the Fox paper in the discussion.

Page 1, Line 18: "...effectively no impact on reducing intra-sample age reproducibility" Why would it? We already know what the major causes for intra-sample grain age variation are, even though they are poorly characterized on a routine basis. Even though grain characterization 'errors' have always been a known problem, we know that 2D estimation isn't that bad, otherwise He ages would never work out correctly during FT correction. Well-behaved whole grain samples show this well and you demonstrate this here that for age calcs. it all works out. The magnitude of age dispersion due to inclusions, zoning, or especially 'anomalously sited' 4He far outweigh 2D grain measurement-derived errors for FT correction (see recent papers by Idleman et al.

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2018 Chemical Geology or Mcdannell et al., 2018 Geochimica et Cosmochimica Acta
–the latter more directly deals with causes of age dispersion)

Response: We motivate this study in the last paragraph of the introduction (Pg 3 Line
11). Although the impact of grain measurements on He age errors is 'known' to not be
'that bad', there hasn't been a quantitative systematic evaluation of just how much error
it contributes to the He age of a sample. Evans et al 2008 and Glotzbach et al 2019
are the only other studies that compare 2D and 3D grain measurements. Our study
builds on this previous work in several ways: 1) previous work has used a of max 24
grains, whereas we use > 100 for a more statically robust analysis. 2) No previous work
actually dated the grains after characterizing them. Here we collect the aliquot ages
and calculate the mean sample ages for the entire population and by subsampling the
data (the latter added upon revision). 3) We show that a 5 μm resolution is sufficient
for CT scanning characterization, which enables many more grains to be measured at
a time (100's). 4) We present a CT mounting and scanning method + new software
to efficiently and cost-effectively collect 3D measurement data for (U-Th)/He. 5) We
highlight that 2D measurement error can have a much larger effect on eU and ESR,
which are ultimately often necessary for making sense of dispersed (U-Th)/He data.

Page 2, Line 11: Why would someone 'assume' parent nuclide heterogeneity without
any proof of it?

Response: We say this because most (U-Th)/He measurements are done on whole
grains without complimentary measurements to check for parent nuclide zoning. In
these cases, the corrections + (U-Th)/He age are calculated with the assumption of
homogeneous parent nuclide distribution.

Page 3, Line 23: Number of grains? chi-squared? % dispersion? all informa-
tive/useful...because that is a highly precise AFT age.

Response: Surpless et al., 2002 report a P(X2) = 75.4%, and measured 25 grains and
100 track lengths. Text to this effect has been added to the manuscript. Full analysis

information are of course also available in the original papers.

Page 18, Line 15: If it is old compared to the rest of the grains then it must contain anomalous He content. Personally (IMHO), I don't like these types of 'catch-all' statements in papers dealing with unexpected He age data. Rather than the usual retelling of the laundry list of potential issues such as 'He implantation, mineral inclusions, fluid inclusions, U-Th zoning, etc'. - just make a general statement that the age was 'bad or unexpected' - you don't actually know why. If you don't actually have any way of quantitatively assessing the reason then speculation is not useful. By listing the many issues it subliminally perpetuates the idea that there are so many problems that we don't know where to start or what to do to fix things. Page 19, Line 6: You should stress here that labs should not pick grains with inclusions regardless. It was just done here to illustrate that the CT scan can pick up fluid/mineral inclusions, right? This text as-is makes it seem like it is okay to date as long as they aren't U/Th-bearing inclusions (which you wouldn't know until it was too late).

Response: We write that implanted He "potentially" contributed to this outlier, but that the reason isn't evident from our data. We've removed the 'potentially due to He implantation' part.

Please also note the supplement to this comment: https://www.geochronology-discuss.net/gchron-2019-3/gchron-2019-3-RC2- supplement.pdf

Response: We have gone through the supplement and made edits according to these suggestions as well.

Please also note the supplement to this comment:
https://www.geochronology-discuss.net/gchron-2019-3/gchron-2019-3-AC2-
supplement.pdf