Response to referee comments

RC1: Comment on egusphere-2026-1552 – David Chew

This is a great paper, showing a really nice integrated methodology for in situ U-Th/He, FT and U-Pb dating of apatite in Teflon mounts. It is going to be a valuable contribution to the field.

I think what would be useful for this manuscript is a figure comprised of a series of schematic panels illustrating all the steps in the triple dating and also explicitly stating the order of the analysis steps in the abstract - mounting, etching, grain selection; FT counting, H measurement, volume estimation and then LA-ICP-MS analysis.

Response: We agree with the reviewer and have created a figure illustrating the analytical workflow and provided it in the abstract.

I could not download any of the scripts from the Zenodo repository – were they meant to be visible to the reviewers? It would have been useful if they were.

Response: We are sorry to hear that the download was not possible. We unfortunately didn't check if the repository is accessible at the time of submission. Access is now open to the public.

My only reservation is the U-Pb age calculation. A zircon should not be used as the U-Pb age standard, and Durango is far too young for an accurate correction. McClure Mountain was analysed – it should be the primary. Why was it not used? You also cannot perform a drift correction on the 206Pb/238U ratio because the standards invariably contains variable common Pb – the common Pb correction must be undertaken first, and then you drift correct the common Pb-corrected 206Pb/238U ratio. See Chew et al., 2014 Chemical Geology which discusses all this in detail. Minor edits are listed below.

Response: Thanks for the comment on the U-Pb calculation. We do agree that the applied analytics could be better for this part. We have not used a zircon, this was still in the manuscript from an earlier version. We deleted it in the revised version. Although we are now routinely doing so, we have unfortunately not measured McClure Mountain apatite in that session. We therefore have only Durango apatite as reference age standard, which we agree is not ideal. We have followed the right order of data reduction, we first applied the common Pb correction for the used age standard, in our case Durango using the 207Pb correction method. Afterwards we corrected the samples for drift. We changed the text accordingly to clarify the order of steps in the U-Pb calculation.

Best wishes

David Chew

Minor comments

L46 'and expanded' – *Have been changed accordingly.*

L51-52 Recent progress in in-situ (U–Th)/He thermochronometry has demonstrated its compatibility with other dating systems, such as U-Pb, Lu-Hf, and fission-track methods (e.g., Carrapa et al., 2009; Bedoya et al., 2024).

I do not get this sentence. Why does compatible mean in this context? Surely the point about in situ (U-Th)/He is that it works (i.e. it reproduces conventional single crystal analyses of age reference materials) and it gives you spatial control. – *We wanted to point out that the in situ (U-Th)/He method is capable to be combined with other dating methods. We adjusted the sentence in the revised manuscript.*

L61-63 A more recent innovation by Bedoya et al. (2024) involved a sequential analysis of Lu-Hf, U-Pb, and after polishing, the LA-ICP-MS-based fission track dating method was applied to single apatite grains embedded in epoxy mounts.

Emphasize the point here that it does not require extraction from the mount. Is the analysis order correct? It would seem to make sense to count for FT first before the grain is more or less destroyed by the big Lu-Hf spot. – *We will emphasize that grain extraction is not necessary. In fact the exact order or analysis is unfortunately not given in Bedoya et al. (2024), but we will try to get this information by contacting the author and correct the text accordingly. OK, we got a response, they first did FT, followed by U-Pb on the same spot. The much larger Lu-Hf spot was placed next to it, in case grains were large enough. I added this information to the revised manuscript.*

L64 Since epoxy degasses under ultra-high vacuum conditions

Is this true of all epoxy resins? Has anyone tried Petropoxy under a ultra-high vacuum? – *I am not aware of any testing on different types of expoxy. We revised the sentence to say that the commonly used epoxy degas in ultra-high vacuum.*

L105 1. Initial Setup: A clean glass slide was coated with a thin film of epoxy to fix grains in place during mounting.

Would double-sided tape work – presumably the epoxy will cure at room temperature leaving not much time for grain placement. What epoxy was used? – *We have tested heat-resisting tape (Kapton), but it was shrinking at our embedding* *temperature of 295°C. We therefore used epoxy resin (Araldite 2020) to keep grains in place. We have added this information in the revised manuscript.*

L164 The zenodo link does not appear to work, and in any case it is not clear what of the following six numbered steps it is used for. – *The Zendo repository contains a Python macro that is semi-automatically helping the user to do the mentioned steps, e.g. switching between objectives, taking images and saving images in a folder structure. We added a sentence to make clear that the macro is guiding the user through the six steps.*

L199, L234,L272, L301 These zenodo links do not appear to be valid either in the DOI system. – *This has been solved in the meantime.*

L301-312 – see substantive comment.

L322-323 I do not like the term variable common lead composition referring to Durango. I think it is accepted its initial Pb isotopic composition is homogenous (see Paul et al. Chemical Geology 2021); what varies is the amount of common Pb in each analysis – so refer to variable amounts of common Pb, not variable Pb composition. It is an important difference. – *Thanks for pointing this out. We have changed the text accordingly in the revised manuscript.*

L418 The IM field is a bit broader that – it includes low ASI (non-peraluminous) granites. There is a pre-IOA rhyolite along with pre- and post IOA ignimbrite in Durango, and the the Durango-Mazatlán ignimbrites are true rhyolites in composition that follow a high-potassium calc-alkaline trend (McDowell et al., 2007). So they would likely plot in the IM field. – *Thanks for clarification, we have added this information to the revised manuscript.*

L449 I would present these data as a lower intercept age on TW concordia – it is very unusual to present 207Pb-corrected data on Wetherill concordia as it makes everything perfectly concordant. Same for sample 22BGE055T in Fig. 4. – *Deriving an intercept age for the Durango data is difficult since the spread in common lead is very low. Therefore we used the y-intercept of 0.862±0.004 (Paul et al. 2021), resulting in a reasonable intercept age of 31.2±3.5 Ma for Durango1. Fitting the 22BGE055T sample in* *the TW concordia is not ideal, the data does not follow a linear trend, which is required to fit a meaningful trend line to derive a lower intercept age. We changed the figure and also reported the newly derived lower intercept age for Durango in the revised manuscript.*

L630-631 What inverse thermal history modelling software was used – your own or HeFTy? Needs more detail. – *Since HeFTy cannot yet handle in-situ data we have our own Matlab based inverse modelling approach that is using standard annealing model of Ketcham et al. (2007) and the adapted He model of Glotzbach and Ehlers (2024). We added some details on the implementation in the revised manuscript.*

RC2: Comment on egusphere-2026-1552 – Rebecca Flowers

This manuscript presents the methodology of the Tubingen lab for the acquisition of laser-ablation U-Pb, fission track, and (U-Th)/He data in phosphates. It presents results for the Durango apatite standard as well as for a sample of crystalline basement in western Germany. It also outlines a decision matrix to enhance the acquisition of high-quality laser-ablation (U-Th)/He data and presents thermal history modeling results for the western Germany basement sample.

Overall, this is a nice paper and I greatly appreciate the overview of the methods in this manuscript. Given that laser-ablation (U-Th)/He dating methods are still under development and there is no universally adopted approach, I agree that it should be a priority for labs to clearly explain their methods prior to publishing data for unknowns. Papers like this one are valuable for moving the method forward.

Our lab at CU is also deep in the development of our analytical methodology for laser-ablation (U-Th)/He geochronology so three of us (myself, Cullen Kortyna, and Jim Metcalf) discussed this manuscript. I summarize below the key points of our discussion and our suggestions to improve the contribution.

Methods

We appreciate the details provided in the methodology. However, it would be helpful to locally provide more information, explain the rationale for some decisions, and include pictures (rather than solely illustrations) of some of the method steps.

- It would be helpful to show pictures of the mount preparation process. We were unclear what was meant by the "heated press". Does the Teflon mount remain on the glass slide or is it popped off for polishing? How do you successfully polish a Teflon disk this thin? – *The details were added to the revised manuscript and we also did a picture of the relevant instruments/parts. We do have a manual heat press like this https://de.kinteksolution.com/products/manual-heated-hydraulic-lab-press-with-integrated-hot-plates-hydraulic-press-machine. The Teflon will be removed from the glass slide and attached to a thicker mount with double-sided adhesive tape for grinding and polishing.*
- It would be helpful to provide more explanation about how you determine pit volumes. We have learned that LA-(U-Th)/He results are very sensitive to the pit measurements and different systems work differently. How does your confocal laser system work to generate models of the pit? What is being used for the top surface interpolation? – *That is a very good point and we are unfortunately missing standards that can be used to validate our measurements. Our system is scanning the pit at vertical resolution of 0.4*

micrometer and builds the model based on where the laser is focused (highest intensity). The pre-ablation surface is linearly interpolated from numerous x,y,z points taken a few microns around the pit. In fact we draw a circle around and take the x,y,z points from that circle. We were also not sure how accurate our method is and for that reason we used step height standards from AppNano with heights of 5 and 10 µm. With our system we could measure the reference height within 1-2%, but that difference might be due to height variations of the reference material. Therefore we cannot really assign an uncertainty based on the comparison with the reference height standard. However, we are very confident that our system is accurately measure the steep walls of the laser pits, since we very accurately determine the vertical wall of the step height standards.

- Uncertainties on pit volumes are not discussed. How did you determine these uncertainties and what is the typical uncertainty magnitude? The pit-volume uncertainties reported in Table 4 are extremely small. – *Initially we have repeated the pit volume calculation several times and calculate the mean and standard deviations from several measurements. In some cases results were very robust and did not change much, in other cases there was a bit of variation, mostly when for instance the grain surface was not perfectly smooth. Anyway, uncertainties were quite small and we therefore did not continue to do repeated pit volume measurements. We do think that a more detailed study is required to study uncertainties of the pit volume measurements. We have added a paragraph in section 2.8 'Pit Volume Estimation' how we did the height calibration of our system.*
- We were surprised that the U-Th spot was placed as a smaller spot in the He pit, rather than as a larger spot over the He pit. The latter would yield improved precision on the trace element data and better sample the parent isotopes that contributed to the measured He. Could you explain the reasoning for this decision? – *We added in the revised manuscript details on our decision. I do agree with the reviewer, but there are also advantages doing a nested approach that we describe. First, the pit shape and geometry will be more similar to those of the glass standard when starting with a flat surface with the larger He pit. Second, potential zoning might be easier to detect and used for age calculation since the ablated material can be transferred into depth information.*
- How was 204Pb corrected for 204Hg for plotting on the Wetherill diagram? – *We have used the 207Pb or 208Pb method to correct for common lead, which has been added in the revised version of the manuscript (section 2.11 U-Pb age calculation).*
- We were unsure why 91500 zircon was mentioned as an age standard when apatite samples are being analyzed. – *Ups, that got probably in the manuscript during an earlier phase of the manuscript. At that time I thought of putting also some zircon data into it. We have deleted it.*

- We typically run a secondary U-Th reference like 610 to check for bias in our calibration curve and wondered if this was part of your standard protocol. – *That is a good point, so far we have not done. Since our Durango apatite yield correct ages, we did not thought to analyse another trace element standard. We agree, however, that it is low effort to measure in addition to the 612 standard. We will follow your suggestion from now on.*
- We see that variable laser parameters (e.g., variable fluence) were used for some of the analyses and wondered if this affected your dates. – *Based on our limited number of measurements with variable fluence (1.3 to 3 J/cm2) we do not see any impact on resulting He age (Table 4). We added this information in the revised manuscript.*
- There are sections on fission-track age calculation and U-Pb age calculation, but no section on (U-Th)/He age calculation. The latter should be added. – *We added a corresponding section.*
- In the section on (U-Th)/He age calculation, it would be helpful to include a section on uncertainties (including those for the pit volumes) and how these were propagated into the (U-Th)/He date. What was the density assumed in the age calculations? – *That is a good point, we will add information on how we did propagate uncertainties and give details on the density used.*
- I would recommend switching the order of 2.10 and 2.11 so that they are in the same order that the results are presented. – *Good point, we changed the order to fit the order in 'Results' section, first U-Pb, followed by fission track and (U-Th)/He last.*

Results

- We appreciate the modeling of mounting-induced annealing and diffusion. We agree that this analysis needed to be done and it is reassuring to see that the mounting protocol does not bias the analytical outcomes. – *Thanks.*
- There is no graphical depiction of the LA-(U-Th)/He dates in the manuscript. Please include results plots for the (U-Th)/He data. These could be data distributions or date-eU plots. - *We decided to add a figure showing the relation between (U-Th)/He age and eU (new figure 5).*
- Lines 513-522 – could these data define a date-eU relationship? Include this plot? – *As is shown in figure 5, there is not trend in date-eU for that sample.*
- Table 4. Please explain the numbers in last four columns – I understand that these likely refer to the decision matrix. – *We added a sentence to section 4.5 to refer to the exclusion criteria.*
- We were not convinced by the interpretation of the U-Pb data for the sample from Germany. The data in Figure 4C on the Tera-Wasserburg diagram

arguably define two different concordia lines, possibly due to two different common Pb compositions. Why not fit one or two concordia lines through these data and derive a preferred date? – *We explored this in detail and fit one/two discordia lines and evalulated their fit using the BIC (Bayesian Information Criterion). Fitting two concordia does yield a higher BIC and is therefore statistically unlikely. The absence of a clear linear trend in the data, however, we also do not want to fit a single concordia and therefore do not fit the data in the Tera-Wasserburg diagram.*

- What type of 204Pb correction was applied to the German sample data on the Wetherill diagram? – *We applied a 207Pb correction method, which we mentioned in the revised version of the manuscript.*
- The large spread of data on the Wetherill diagram in Figure 4D suggests that the common Pb correction was not successful – this would indicate that taking the average 206Pb/238U age or the weighted concordia age is not the best approach to deriving an apatite U-Pb date for this sample. Instead, the intercept from the Tera-Wasserburg diagram would appear to be a better approach. – *As mentioned above, fitting the data in the Tera-Wasserburg diagram is hampered by the observed spread and non-linearity of the data. This was mentioned in the text and we also added a sentence in the figure description. After applying the common lead correction, most measurements overlap in the Wetherill diagram.*
- The authors provide geological explanations for the discrepancy between the K-Ar date and the apatite U-Pb date. It strikes us as more reasonable to question the approach to the common Pb correction, and we are generally skeptical of the quality of K-Ar dates from the 1970s. It seems to us more appropriate to disregard the K-Ar data and then derive a U-Pb date from the Tera-Wasserburg diagram (and not worry about it being even older than the date currently derived from the Wetherill diagram). – *We were a bit defensive in interpreting our results since the sample did not produce ideal results. As mentioned above, the scatter of the data in the Tera-Wasserburg diagram does prevent the derivation of statistically meaningful intercept age(s).*

Discussion – Decision matrix

- We feel that the decision matrix is a valuable contribution of this manuscript. However, we feel that there should be a richer discussion of the four different parameters in the context of the actual data. How do the dates reported in this manuscript correlate with these different criteria? It would be helpful to show plots of the data in the context of these criteria. – *We added a few sentences to discuss the impact of the laser pit shape and especially the radionuclide zoning on the resulting ages and uncertainties.*

- Here again it would be helpful to include real images, such as of the laser pit shape. – *We have created a new figure, the same style as the decision matrix figure but with real images of grains. We will put this figure into the supplement.*
- We like your statistical approach for radionuclide zoning, but could you please explain in the text why you decided on 20 as the MSWD cutoff? – *We had a closer look on our radionuclide variations with measurement time and compared the measured core-to-rim ratio with the modelled effect on whole-grain ages. A MSWD value of >20 roughly correspond to a >2 step-like variation in radionuclides and, according to our estimates to an age departure of >10% (Glotzbach and Ehlers, 2024). We added this information to the revised manuscript.*
- We were confused about the grain geometry criterion – if you put the pits on the grain geometry schematics, isn't this criterion essentially the same as the pit-grain relationship criterion? As one moves from 1 to 5 in grain geometry, the grains become smaller, which means that the pit becomes closer to the grain edge, the same as in the pit-grain relationship criterion. Could you please add the pits to the grain geometry schematics and explain in the text why you distinguish the grain geometry criterion from the pit-grain relationship criterion? – *Good point; we added pictures of the pits on the grains shown in the 'Grain geometry' column in the decision matrix figure. Both criteria are different; while the 'Grain geometry' does not necessarily impact the resulting ages, it does have consequences for the thermal modelling, since the distances to the grain boundaries are partly unknown, preventing to set-up all required modelling parameters (e.g. grain radius and distance to the prismatic surface). The 'Pit-grain relationship' does directly impact the resulting ages, however, thermal history modelling is still possible. We added some extra text to the discussion section to explain the differences in more detail.*

Discussion – Tradeoffs

- We recommend adding a short section in the discussion that discusses the tradeoffs when collecting triple dates on apatite. – *That is a great idea, we will add a section on the tradeoffs that derive from making compromises.*
- For example, the fission-track data are of low quality because the authors restricted themselves to collecting those data on samples also dated by U-Pb and (U-Th)/He (at least, I think that's what was done). Would it be better to acquire data for a larger suite of apatite, even if not on the same grains dated by the other methods? It would be worth discussing the pros and cons. – *We will add information on the grain selection and especially on the restriction of counting in a specific geometry, e.g. circle with 30 µm. We do have*

some suggestions on how this could be optimised, e.g. measuring/counting several spots in larger grains.

- A compromise must also have been made when deciding to collect the rare earth element data simultaneously with the U-Th data. Collecting all of these data will reduce the resolution on the U-Th zonation and increase the uncertainties on the U-Th. – *Integration times of the REE are 0.001 sec/Mass and therefore all 14 REE measured do only take 0.014 sec, while we measured Pb isotopes with an integration time of 0.1 seconds, and the whole sampling period is roughly 1 sec. Therefore, the measuring time of REE is negligible and does not led to a reduced resolution of the U-Th-Pb measurements. We will add this information in the discussion.*
- We fully appreciate that compromises are required when setting up analyses and deciding what data are most important to prioritize. A discussion recognizing these limitations and decision-making would be helpful. - *We fully agree that this needs to be stated and we will add a short paragraph reporting our setup and potential compromises.*

Discussion - Thermal history reconstruction.

- We appreciate the thermal history modeling carried out by this paper and the efforts to derive thermal histories that simultaneously explain the triple dates. We have a few thoughts here.
- Table 5. Please explain the Dist2PrismX and Dist2PrismZ parameters. How to you know distance from polished surface to the grain bottom? – *Dist2PrismX and Dist2PrismZ are the measured distances of the centre of the laser pit towards the prism faces measured in the horizontal and vertical plane. The horizontal distance is easy to measure, the vertical is not trivial and also not very accurate. We simply focused on the bottom of the grain with transmitted light and corrected the distance with the refraction index of apatite. We added this information in the revised manuscript.*
- Lines 652-654. Why use acceptable fits instead of good-fits? – *Ah, of course, we also take good paths, everything that yields a GOF >0.05. We rephrased it.*
- In Figure 7, could you please show a plot of all the LA-(U-Th)/He dates (not just the 7 modeled dates) along with the predictions of the best-fit path? This could be a date-eU plot, or some other plot. A plot comparing all observed dates with the predicted dates would be far more intuitive than the plot currently included, which shows the observed and predicted concentrations (which carries no geological or kinetic meaning). - *We generated this plot, and replaced the subfigure 7A with the new plot.*

Minor Comments

- There was a lot of space and two entire figures on the modeling of mounting-induced annealing and diffusion. You might consider moving some of that content into the supplement. – *We do agree, and accordingly slightly condensed the section on the 'Mounting induced annealing and diffusion' and move the original Fig. 1 into the supplement (now Fig. S2).*
- Lines 28-29. I think too many significant figures are reported here? – *Note exactly sure what you ment, but we we deleted '(aliquot 1)'.*
- Lines 218-219 say 20-50 um spot size for He pits, but lines 269-270 say 30-50 um spots. – *Ah, 30-50 µm is correct and was corrected in line 218-219.*
- Line 724. "Resolute" doesn't seem like the right word choice here. "…better-resolved cooling histories…" would be an alternative. - *Thanks for the suggestion, we will correct this sentence in the revised manuscript.*