## Revision

## **Associate Editor Decision:**

**Publish subject to minor revisions (further review by editor)** (02 Mar 2021) by <u>Axel Gerdes</u> Comments to the Author (pdf): <u>gchron-2020-19-comments-to-author.pdf</u> Comments to the Author: Dear Bar Elisha et al.,

my apologies that this response comes so late. I am really sorry for this...

One reviewer and I have elevated your revised manuscript. While it has improved in several parts, we both felt that the effort in revising was not sufficient. there are several suggestions and issues raised by the reviewer which you more or less ignored.

Please find additional comments/suggestions by one reviewer and by me (below or ass attachment). I would like you to carefully read them and hopefully, this inspires you to do some additional modifications to your manuscript. The reviewer and me, both agree that the ms is still poorly structured and lacks a clear message of what is your new finding. Note that the comments from both of us aim to improve your manuscript.

All the best Axel Gerdes

Dear Axel Gerdes,

We very much appreciate the comments and suggestions of both you and Jon Woodhead's and performed the following modifications accordingly:

In this revised paper we changed the basic structure of the manuscript so that the results and the discussion are now presented in two separate chapters. We now present the results in a straightforward manner without interpretation and then discuss the significance of the results with respect to the expected ages. We continue the discussion by presenting further investigation of textural characteristics of the studied dolomites that might affect the resulted ages.

In the revision we acknowledge the notion that ablation efficiency could not be the main cause of the discrepancy between obtained and expected ages, however, we did not rule out the possibility that multiple imperfections in the morphology of laser pits in micritic dolomites effect at some extent the 40–70% age offset obtained in samples MAM-3 ad MAM-7. We rewrote this section to focus on different mechanisms for age mixing, and as suggested by the reviewer, we pointed out the possibility that detrital contamination may lead to ages older-than expected. We further focused our discussion on the option that mixed ages are due to chemical zoning of the crystals and due to grain size smaller than the spot size of the laser.

Minor additional modifications and clarifications are incorporated in the revision and are described in red text following your comments:

my comments:

Line 15-16: This sentence is incorrect. You do not show/prove that dolomite dating is sensitive to ... "you only discuss this!!!

We changed the sentence as followed:

"We show the complexity of *in-situ* dolomite dating and discuss variables such as crater morphology, textural differences, chemical zoning and detrital impurities that may affect the interpretation of the resulted ages"

Line 18-20: make two sentences out of this as these are two things. From my point, you overestimate the necessity of a perfect matrix-matched standard. As your data shows it mixing of different phases during laser ablation is/can be the dominating effect, which yields results that are totally wrong. It hasn't been yet explored (be fair on this and not biased by your opinion) how much inaccuracy non-matrix matched standardization caused in the case of dolomite (No, the paper of Guilliong et al., not has proven this!! it only suggest it).

We changed the sentence as followed: "We conclude that age mixing at the sub-millimeter scale is a major challenge in dolomite dating that need to be further studied. We also note the importance of matrix-matched standards for reducing uncertainties of the dated material."

Line 38: you citing 5 papers that applied U-Pb dating also to dolomite and come with a sentence it has been "mostly applied to calcite". It confuses the ready a bit. We change to: "Recent developments of LA-ICP-MS has opened a new avenue for measuring

absolute ages of carbonates, thus improving the understanding of many fundamental geological processes, such as ..."

Line 46: Guillong et al. do not provide a thorough methodology of dolomite dating, this is wrong. He might suggest/discuss that this is needed. Just as information, we tried to reproduce Guillong et al. experiments and failed. His approach yields misleading results. There is an effect but it is much smaller.

We changed the sentence as followed: "While LA-ICP-MS analyses on calcite evolved to be a conventional method of dating (Roberts et al., this issue), a thorough methodology for dating other carbonates, such as dolomite, is still needed (Guillong et al., this issue)."

Line 74: MSWD: write out what it means when you used it the first time We added "Mean Square Weighted Deviation (MSWD)"

Line 77: This is unclear, Hg contamination?? -> Avoid difficulties related to Hg interference correction on 204Pb.

We rephrased as followed for clarification: "The <sup>204</sup>Pb concentration was calculated using the <sup>206</sup>Pb concentration and assuming a Stacy-Kramers <sup>206</sup>Pb/<sup>204</sup>Pb ratio to avoid difficulties related to the Hg interference on <sup>204</sup>Pb."

Line 65-72: as your error ellipses are rather large considering using an MC-ICPMS you should mention the error propagation you used. I assume you added 1% and 5% uncertainties to your isotope ratios. It is hard to understand for many people, why you do this. and this is important information as it might be one of the reasons why your MSWDs are below 2, even when you seem to analyze "mixtures"... I only want you to think about it, but adding this information is crucial !!! and a short comment to me would be helpful to understand.

We added the following comment for clarification: "Uncertainties were propagated on individual unknown ratios such that <sup>207</sup>Pb/<sup>206</sup>Pb (2%) and <sup>206</sup>Pb/<sup>238</sup>U (4%) ratios of a zircon standard—run throughout the session (Mud Tank; Black and Gulson, 1978)—yielded a single population; this resulted in reasonable MSWDs for the calculated ages of calcite RMs"

Line 168: error: the correct expression would be uncertainty and I suggest that you use it from time to time.

Yes, we have changed throughout to uncertainty

Line 168-170: "large error" is relative and you use it in the text for uncertainties of 25% and as well as for <10%. As one can have uncertainties of even 100% I would speak rather from moderate to elevated uncertainties for the range of 10-30%. We agree and changed to "moderate to elevated uncertainties"

Line 170: "Data point analytical uncertainties are smaller than the scatter of the spot analysis ...." ??? this is misleading and "nonsense"... your uncertainties are elevated and the total range is very small, so it is not a surprise that you are getting a good correlation with low MSWD. First, it is misleading and incorrect to call it an isochron, secondly as everybody knows a low MSWD can result from an overestimation of uncertainties.

This sentence was removed, as it provides little insight.

Line 186-190: As also outlined by the reviewer, this is not correct and wrong. Guillong et al. imply that you can have an inaccuracy of 4-8% due to different ablation efficiency but not 40-70%. You also not show that your ablation volume is so different. So ablation efficiency can have only a minor effect on the inaccuracy of your data but the main effect is the mixing of different materials (formed at a different time). It is confusing for the reader that you seem to favor both processes equally. But it is incorrect and missing any scientific evidence that you can explain this with ablation efficiency!!!

We agree that there is little convincing evidence that the ablation efficiency could be the cause of the discrepancy between expected and obtained dates in this study. Therefore, we rewrote this section stating first that ablation efficiency is unlikely the main factor in the age discrepancy, and later pointed out some minor differences we found. However, we mentioned that the observed morphological differences in laser pits of micritic samples MAM-3 and MAM-7 might affect at some extent the 40–70% age offset we obtained. We now included the possibility that detrital contamination may lead to older-than expected ages and further focused on the problem of mixed ages due to chemical zoning of the crystals and grain size smaller than the spot size of the laser.

Line 192: the age of 93 +-7 Ma is not slightly younger!! This is incorrect, It is within error!!! This is one of several cases where it seems that you want to influence the reader. Overall I am missing an unbiased presentation of the results. You struggle with the interpretation of your data set (I

understand, it is not straightforward) and feel the need to interpret it as it would strongly imply one or another idea. But as you make two processes responsible, the unexpired reader stays confused as the discussion is overall confusing and not so well structured (jumping from one to another). The ms would improve by a more careful language/discussion, by weighing possibilities better and suggesting possible interpretations instead of implying one and later another.

We agree with your comments and therefore rearranged the ms accordingly so that the Results and Discussion are now two separate sections. We rewrote the results section and removed sentences that might influence the reader. In the Discussion section we discuss the significance of the results (section 4.1) and then discuss textural characteristics that might affect the results (section 4.2).

Line 210-235: section 3.2: adding this not make the case stronger. You rather confuse the reader more, by repeating what is written before and after... the effect of ablation efficiency is to my knowledge overestimated and importantly you have no hard data to add much to this discussion. Mixing of material is a much more likely interpretation of what you show.

Sections 3.3 to 3.5 are the important ones for me, and the reader gets confused by extending too much the discussion of the ablation efficiency ...

Please see previous comments.

Line 362: Careful, this is not an observation you can make based on your data! We changed this to: "A comparison of down-hole fractionation between RMs and unknowns, even those of similar chemical makeup, can be a valuable tool in estimating true uncertainty and inaccuracy of unknowns."

Line 364-365: Careful, this is not correct. I can't see this supported by your data!!! Delete this sentence or re-write.

We changed to: "Textural characteristics such as micritic vs. well-crystalized grains have minor effect on ablation efficiency and can have only minor effect on the resulted ages."