Geochronology Discuss., referee comment RC2
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Comment on gchron-2021-27

Anonymous Referee #2

Referee comment on "Calcite U–Pb dating of altered ancient oceanic crust in the North Pamir, Central Asia" by Johannes Rembe et al., Geochronology Discuss., https://doi.org/10.5194/gchron-2021-27-RC2, 2021

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Although the manuscript is nicely written and I like the idea of dating Palaeo oceanic curst by U-Pb in calcite, I can't recommend a publication in its present form. I see an overinterpretation of the analytical data and therefore recommend a cautious re-evaluation of the ages and their significance. I refer mainly to the analytics used, the assessment of the precision and accuracy of the data, the presentation of the results. The data must be presented more completely in the tables (online), the standardization and error consideration must be explained more clearly and adapted to the standard procedures currently in use.

I do not believe that the calcite ages match those of the ocean crust, but possibly within an age error/scatter range of 3-6% (which I believe is realistic for the data). Be aware that it is an interpretation of the data, it could be also the result of the mixing of a complex domain consisting of multi-generation veins. I would suggest using an age range (e.g. 310-330 Ma) for each sample as the results do not suggest that they formed during one short-lived event. Alternatively use weighted average (carefully evaluate the uncertainty, see below), although it is statistically incorrect.

- Method

Referring to Su et al 2020 and Yang et al 2021 for a description of the method is insufficient, both papers are also application papers. You need to explain how the raw data were treated; corrections and outlier rejection, standardization, and uncertainty propagation have been done. This has to follow the recommendation of Horstwood et al. (2016, Geostandards and Analytical Research)

Data in high-rank journals should include comparable data sets, in which uncertainties were correctly propagated (see Horstwood et al. 2016): random uncertainties, from background correction, counting statistics and access of variance (drift correction using NIST614), and systematic uncertainties, ratio uncertainty of RM (>1%), long-term variance (>1% !), decay constants.

It is not clear how the authors correct for matrix-related Pb/U fractionation (using one or both of the mentioned carbonates). The material they used is no international reference material and its age and uncertainty have not been evaluated by an independent method (e.g. ID-TIMS). Please explain in more detail the applied approach to re-evaluate your uncertainties and uncertainty propagation. Report data in table S2 of your reference material measured during the sessions, report data for long-term variance (reproducibility) of your method and that of access of variance of your NIST614.

The authors use WC-1 (incorrectly labeled WC01 in their text) as secondary RM to evaluate the accuracy of their data. However, based on this the data is up to 5% inaccurate. The data of Roberts et al. reflects some heterogeneity of this material and is explained by sampling bright and darker domains to achieve some spread in the Pb/U (see Rasbury et al. 2021). They report a 2.5% uncertainty. Rembel et al. report a 1% younger age with a 1.5% uncertainty (no systematic uncertainties propagated), which means the data can be up 5% inaccurate! This needs to be mentioned in the paper and considered in the discussion of the data!

Based on the data it is not possible to evaluate the precision and accuracy of the ages. However, the authors report ages in the text of the manuscript with a precision of better than 1%. This is misleading and gives the impression they can date their sample and events with precision and accuracy of better than 1%. For the majority of carbonate ages so far published, uncertainties range from 3-5% or worse (Roberst et al. 2021, Geochronology) and only very few labs demonstrated a long-term variance of 2% (e.g., Guillong et al. 2021, Geochronology). The authors need to access their precision/long-term variance/accuracy and should not quote any uncertainties in the text with the figure behind the dot (300 +/- 4, instead of 300.5 +/- 3.6 Ma) and with no or maximal one figure (!!) after the dot in the figures.

U-Pb ages: Fig.4: First of all, I can’t reproduce exactly the ages using the data presented in table S2. Secondly, an MSWD of around 2 or higher means the data forms, not a uniform population/event or uncertainties were not probably propagated. In the first case, it would mean the different areas are not formed at the same time. In figure 2 it is shown that data was acquired from different domains, so you can’t group them together and calculate a common age as you have done. This is a mixed-age with misleading incorrect precision and accuracy. You should report the ages of individual areas and compare these ages with each other as you have done in figure 3. So change Fig 4, only use some representative examples for the Tera-Wasserburg diagram. If you want to use a weighted average, do not forget that they likely represent mean ages, representing more an age range. But still, you have to add systematic/expanded uncertainties to the final age on the weighted average ages when comparing it with other data. So for Fig3 weighted average use 316 ± 8 / 12 Ma (internal/expanded uncertainty).

As it is visible from scans B3, B4 the investigated domains do not form by a single process of calcite vein-forming but show a more complex pattern with different generations of cross-cutting fractures. And still, the authors what to interpret the U-Pb ages as representing a single calcite precipitation event at the time of basalt-crystallization? Please be more cautious in the interpretation of your data. The obtained ages of the different areas of one sample scatter and reducing the uncertainties by statistical tricks using all data in one single Tera-Wasserburg, is not the way to go.

I also have to comment that the geochemical data is not presented very convincing, I would like to see plots showing that the spots of the different domains show similar trace element composition e.g. using elemental ratio plots, incompatible/compatible. Figure caption of D1 does not explain the greyish field (whole rock data??). Having insitu Sr isotope data would support the interpretation.

Line 181-184: A rough overlap of U-Pb carbonate ages with metamorphic (meta-andesites) and felsic magmatism in the range of 350-314 Ma, does not prove that the obtained ages reflect calcite precipitation related to oceanic crust formation.

The U-Pb data tables and metatables have to be prepared accordingly to Horstwood et al., to enable readers to understand the data quality and the method. Data tables should include U and Pb content calculated, the signal strength of 206 and/or 238 in cps, the Th/U ratio, and the rho value (even it is close to 0!).

Samples should be clearly separated in Table S2. Sample coordinates should be included.

From the existing data table S2, I get similar ages and similar uncertainties but not the same numbers as reported!

Figure C1, please include MSWD of each age, add the information that you used 2sigma uncertainties

Line 149: ‘they overlap within 2s-error for each sample'. Please explain this better

Line 169: … ‘overlap mostly within 2s-error per sample’ – I don’t agree with using this as evidence they formed during one event. No, they differ in age, and looking at the scans in B4-B4, it is not compatible with the idea of one single event of vein formation.

Fig. 3: Quote the MSWD for each sample, they do not form a homogenous population, be careful to interpret this as one age. Use in addition the expanded uncertainty (see below/above). The intial Pb of the different domains scatter also quite a bit…

Fig.4. figure caption, explain better, .e.g all data obtained from the different areas of the 4 samples. However, I do not agree that you can plot (U-Pb) them in this way together (see above).

These REE plots are for me not very convincing to support that the veins form during a single event…

Fig.5. Report all the ages with reasonable accuracy (including Ar-Ar ages, LA zircon ages, and the LA carb ages), so without figure behind the dot (only ID-TIMS data should be presented with sub-% accuracy!). It also makes ages not better accessible if more figures are reported.