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Comment on gchron-2021-10

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Referee comment on "Exposure dating of detrital magnetite using ^{3}He enabled by microCT and calibration of the cosmogenic ^{3}He production rate in magnetite" by Florian Hofmann et al., Geochronology Discuss., https://doi.org/10.5194/gchron-2021-10-RC2, 2021

General evaluation

Hofmann et al present here a new pre-screening method based on microCT as a mean to identify the amount and nature of solid inclusions in magnetites. These minerals were collected from a 1.75 m vertical profile below a geological surface that had been exposed for about 54 ka at Earth's surface. Analyzing the helium isotopes released by melting these prescreen magnetites, they perform a whole inventory of the ^{3}He and ^{4}He present in these minerals and compare aliquots that bear inclusions with those that are inclusion-free. Authors notably show that the presence/absence of these inclusions has a major impact on the non-cosmogenic ^{3}He: selecting inclusion-free magnetite thanks to this microCT pre-screening, they show that the inter-sample cosmogenic ^{3}He scatter is reduced. Then, authors compute a spallation ^{3}He production rate (of about 116 at/g/a) from inclusion-free magnetites, after cross-calibration with cosmogenic ^{10}Be measured in the same vertical profile. This production rate is both in agreement with the previous value reported in iron oxides by Kober et al (EPSL, 2005) and the up-to-date value of 122±12 at/g/a reported for the commonly used olivine and pyroxenes phases (see synthesis by Martin et al., QG, 2017, https://crep.otelo.univ-lorraine.fr/#/production-rate).

This new pre-screening method has a great potential since it may be very useful to reduce the scatter of cosmogenic ^{3}He concentrations due to "exotic" ^{3}He production process. Authors present convincing observations and perform the main relevant computations to evaluate and hierarchize the different sources of ^{3}He and ^{4}He in the analyzed magnetites. This contribution will be of interest for the readership of Geochronology. Several issues however need to be addressed during a revision stage, before the manuscript can become publishable. See more specific comments below.

Major concerns

1 - Calculation of (U-Th)/^{4}He* closure ages

To compute (U-Th)/^{4}He* closure ages for all magnetites, authors use a mean U and Th concentrations measured from three different samples only. This is a problem since the inter sample variability of U and Th concentrations may be large and mainly controlled by the amounts of bright inclusions (e.g. zircons), as shown and discussed in this manuscript. I understand that measuring U and Th on the same aliquot than the one used for ^{3}He and ^{4}He analysis face technical limitations, but authors should better acknowledge their assumption of using U-Th measured in 3 samples. They should also better propagate the uncertainty arising from this calculation. Unrecognized U and Th variations may indeed affect the accuracy of the closure age calculation for a specific sample, and also, affect the accuracy and precision of the nucleogenic ^{3}He correction.

Please display the individual (U-Th)/^{4}He* closure age of each sample adding a new column in Tables 2, 3 and 4, or at least in Table 4, the one presenting the unscreened magnetites.

2 - Nucleogenic ^{3}He corrections

The amplitude and the uncertainty arising from the nucleogenic ^{3}He correction should be better presented and discussed, notably taking into account the variance of the helium closure ages due to potential inter-samples and inter-aliquots variability of U and Th concentrations. Adding a column in the Tables with the sample specific nucleogenic ^{3}He corrections (and attached uncertainty) could be useful.

More specifically, the computed nucleogenic ^{3}He contributions reported at lines 348 are not in agreement with the nucleogenic production rates and closure ages given two lines above. Although I find the same nucleogenic production rate using my own code (based on reported major and trace, Li compositions for these rocks), multiplying these production rates with the given closure ages of 15 Ma and 130 Ma yield 0.3 Mat/g and 2.3 Mat/g, respectively (not 1 Mat/g and 7 Mat/g as stated lines 348). Please carefully check these calculations.

3 - Data presentation in the online open access table

The online spreadsheet presenting the data as open format in the NSF website bears mistakes: for some samples, notably 17WW-01 aliquots, reported ^{3}He concentrations are not similar to those reported in the Tables of the Geocrhonology paper. Please check all samples.

Moreover, analytical uncertainties and helium blanks are not presented in this open database. I think they should be provided.

Finally, I find quite strange to report ^{3}He in at/g while ^{4}He concentrations are given in nmol/g. My comment is here about the open database, but also for all sections, figures and Tables of the main article. Why not homogenizing these units?

4 - Citation of many abstract conferences

Authors quote at least 4 conference abstracts (Bryce and Farley, 2002; Cox et al., 2017; Matsumara et al., 2014; Rogers et al., 2013) and one M.S. dissertation (Moore, 2017). Since those materials are not strictly peer reviewed, I wonder if this is compatible with the editorial policy of the Geochronology journal. If not, please, remove these references.

5 – Price of microCT

What is the price and accessibility of such microCT analytical sessions?

Other concerns and suggestions

Line 15: indicate whether this "excess ^3He" is due to magmatic ^3He, inherited cosmogenic ^3He or other sources of ^3He.

Line 25: Give the uncertainties attached to this 53.5 ka ^{10}Be exposure age.

Line 29: “Since” implies causality, and this is strange to use this word here, no? If you agree, I suggest replacing “since” by “while”.

Line 32: Helium measurement may also involve “dangerous task” and use of chemical products. I would remove this quite subjective statement.

Line 34: The necessary amount of quartz for ^{10}Be analysis may be as low as 1 g, in the case of ^{10}Be rich samples (> 10^5 at/g) (e.g. Blard et al., EPSL 2013).

Line 67: “same soil samples…” Quote the previous ^{10}Be study here.

Line 131: Given the uncertainties, 53.9 ka should be rounded to 54 ka. There are too many significant numbers.

Line 137: Did Owen et al., 2014 also compute an erosion rate from the ^{10}Be profile inversion? If so, it can be useful to state this number here.

Line 139-141: State here that you collected 12 samples in this vertical profile.

Line 150: Why disgarding the 100-250 microns fractions? Some studies (Williams et al., QSR, 2004; Puchol et al., Chem. Geol, 2017) demonstrated that the 100-250 microns granulometry bear much less magmatic ^{3}He than larger fractions.

Line 166-167: If possible, why not stating here the correspondence between these inclusions colors characterized by the microCT scan and the real nature of the inclusions?

Line 179-180: Does this statement imply that some elements that are abundant in zircons, such as U and Th, could have been underestimated?

Line 204: Did you analyze the ^{3}He and ^{4}He concentrations in a solid standard, such as CRONUS-P?

Line 250: Why not homogenize ^{3}He and ^{4}He units? Reporting both isotopes in atoms/g (or mol/g) is better.

Line 271 to 276, lines 301 to 304, and Fig 11: 4 crushed aliquots (among 6) have higher ^{4}He concentrations than uncrushed aliquots. Although I agree that inter-aliquots stochasticity may explain this observation, are you sure you can totally discard the possibility of atmospheric ^{4}He adsorption on the surface of crushed aliquots (Protin et al., GCA, 2016)? The probability that this contamination occurred with such an amplitude is directly linked to the new granulometry of the samples after crushing. Can you thus provide a range of the sample size after crushing, at least a first order estimate?

Line 291: “suggests” instead of “implies”. A definitive demonstration would require measuring U and Th concentrations in situ, in these specific bright inclusions, in the same samples used for ^{4}He analysis.

Line 328: State here that these U and Th concentrations are average of 3 samples only, report the standard deviation (that is at least 30%) and propagate this uncertainty into closure ages (if not done).

Line 328 to 330: Presentation of (U-Th)/^{4}He* closure ages: as it is well shown by Fig. 9 and discussed latter, the inter-samples ^{4}He variability is also controlled by the proportion of bright inclusions (zircons) and hence the eU of each aliquot. So, you should better acknowledge and discuss the limitation of your approach that consider mean U-Th concentrations from only 3 samples to compute individual (U-Th)/^{4}He* closure ages.

Line 390: The most up-to-date ^{10}Be world average SLSHL production rate in 2021 is 4.11+-0.19 at/g/a with the time-dependent Lal/Stone factor (Martin et al., QG, 2017) https://crep.otelo.univ-lorraine.fr

Line 397: The most up-to-date ^{3}He world average SLSHL production rate in 2021 is 122+-12 at/g/a with the time-dependent Lal/Stone factor (Martin et al., QG, 2017) https://crep.otelo.univ-lorraine.fr. Contrary to the value reported in (Goehring et al., 2010), this updated P_3 average includes radiogenic ^{4}He corrections and new calibration sites published during the last 10 years.

Line 400-401: In this discussion, the "hence" followed by a "consequently" seems to be a sort of circular reasoning.

Line 405: "large"? What is the typical ejection distance of this reaction?

Line 414: Remove "and CTN" since you only discuss the nucleogenic ^{3}He in this paragraph.

Line 425: Most of pyroxene have Li concentrations lower than 20 ppm.

Line 455: Shouldn't you mention here the impact of nucleogenic ^{3}He?

Line 460: I suggest you mention that this best granulometric window of 400-800 microns is the convolution of two opposites necessity: < 300 microns grains have the lowest inclusion amounts, while 600-800 microns are the biggest contributors of analyzed material (in volume).

Line 465: I suggest adding here than the computed P_3 for magnetite is also compatible with the 122+-12 at/g/a production rates reported for the commonly used silicates (olivines and pyroxenes; Martin et al., QG, 2017) https://crep.otelo.univ-lorraine