Geochronology Discuss., referee comment RC1
https://doi.org/10.5194/gchron-2021-41-RC1, 2022

Comment on gchron-2021-41

Anonymous Referee #1

Referee comment on "Complex ^{40}Ar□/□^{39}Ar age spectra from low-grade metamorphic rocks: resolving the input of detrital and metamorphic components in a case study from the Delamerian Orogen" by Anthony Reid et al., Geochronology Discuss., https://doi.org/10.5194/gchron-2021-41-RC1, 2022

Reid et al. present new mineralogical and ^{40}Ar/^{39}Ar (furnace step-heating) data from fine grained, low-grade metamorphic rocks of the western Delamerian Orogeny, and use that data to distinguish between detrital and neoformed mica components. The data are also used to propose simple thermal history interpretations. The data are valuable, although the data presentation is incomplete, and I suggest that other interpretations can be reached, which have not been sufficiently explored.

My main concern is the ^{40}Ar/^{39}Ar data are interpreted solely considering combinations of the time of mica formation in a source region, the time of recrystallisation in the Delamerian Orogeny, and possible Ar loss via thermally driven diffusion during post-Delamerian tectonics. The logic behind the interpretation is accurate relative to these three issues. However, the authors should also consider the highly significant effects of secondary alteration, especially given the mineralogical data that is presented, which surprisingly is not considered in the interpretation section of the Ar isotope data. A robust approach would be to use mixing lines in ternary space to delineate, for example, between combinations of radiogenic ^{40}Ar, ^{39}Ar(K), ^{37}Ar(Ca), ^{38}Ar(Cl) (although unfortunately the samples were irradiated in a Cd-shielded position). These could be combined with the documented mineralogy (given the lack of microprobe data) to at least draw conclusions about which reservoirs are contributing to each heating step. This is fairly commonly done (e.g. see Challandes et al., 2008; Popov et al., 2019), and avoids ignoring fluid-related effects. E.g., were the protolith, Mesoproterozoic micas not altered at all between their original formation and deposition approximately 500 Ma later? Are the Delamerian-aged metamorphic micas unaltered? Has alteration reduced their age relative to their time of formation?

The ^{40}Ar/^{39}Ar methodology and data presentation are incomplete (see the recommendations in Schaen et al. (2021); https://doi.org/10.1130/B35560.1):

- Line 174 reports that the samples were wrapped in Al foil, whereas line 481 reports they were wrapped in Sn foil.

- Data tables are required for the analyses of the flux monitors and the K2S04 and CaF2 salts. Reid and Forster (2021) only provide data obtained from the samples.
- How was the mass discrimination determined? Provide the values that were used so the dates can be calculated from the raw data by the reader. Were these dependent on ion beam intensity across the range of intensities measured?
- Which collector configuration was used (Argus VI), and what were the Faraday/CDD yields (if appropriate)?
- I gather from the text that the irradiation package included the flux monitors in a linear stack. What was the distance between each flux monitor?
- Provide a description of how the blanks for individual heating steps were determined once the sample was already loaded into the furnace crucible.

Specific points

Introduction: The Introduction is clearly written although it would benefit from some more specific aims that are relevant for the Delamerian Orogeny. E.g., the info in line 108 would be useful in the Intro.

Line 108: Where does this 480 Ma come from? The only previous reference to 480 Ma is in line 76 (Ar/Ar laser analysis of hornblende and biotite; Turner et al., 1996). Are the authors suggesting that the youngest of these Ar dates was previously taken to signal the end of orogenesis? The Ar dates are from granites so surely they just record cooling, and not crystallisation. U-Pb dates would be more reliable. Are there any? Line 69 suggests orogenesis was over at about 490 Ma.

The Hylogger results presented in Appendix A require more clarity. As described in Appendix A, each sample is divided into a series of runs. For each sample, there are separate bands, each of which consists of three rows of coloured squares, which correspond to mineral compositions obtained by either SWIR or TIR. How should these three parallel rows be interpreted? Is each parallel row, one traverse such that each band of three parallel rows is three traverses? Or, is one band of three parallel rows, one traverse? Furthermore, the colour for Mg-Chlorite is extremely similar (almost not distinguishable) from the colour for albite.

Lines 136 – 137. The TIR also shows a considerable amount of muscovite.

Typos in lines 129, 273, 319, 407

The plots in Fig 5 can be improved. Use the full space of the graphs. E.g. in 5a, modify the date axis to range between 200 and 650 Ma (the space between approx.. 750 Ma and 1200 Ma is currently blank).

To help the reader, I suggest colour coding segments of the age spectra and York plots so the reader can match the topology of the spectra with specific trends in the York plots that are referred to in section 5. E.g. line 197. Which final steps, and which points are these on the York plot? Line 213 – which are the beginning and the final steps in the York plot? Etc………

What is the author's definition of a minimum date? In some cases, the minimum date seems to be the 40Ar/39Ar date of the first heating step (e.g. 3779555), whereas in other cases the date of the lowest-T heating step is ignored (e.g. sample 3779551a). If significance is attached to a minimum date, then describe how the minimum date is defined.

Lines 240-242. Theoretically, a range in diffusion characteristics in a single crystal would yield a staircase spectra from a single crystal, depending on the t-T. However, complex age spectra can also be obtained from "single crystals" because they are frequently polycrystalline with perhaps several generations that arise by deuteric and low-T alteration (e.g. K-feldspar from the Chain of Ponds, Chafe et al., 2014; Klokken K-feldspar, Parsons et al. 2013; Itrongay K-feldspar, Popov et al., 2020; muscovite from Larderello, Italy, Bulle et al., 2020).

Line 254. What do the authors mean by "main mineral gas reservoir"? Any combination of reservoirs that don't contain an initial component? There may be several radiogenic reservoirs if the grain is not 100% monomineralic.

Lines 256 – 257. I realise this refers to previous work, but what is the justification for the statement that the older, high-T steps are infected with excess ^{40}Ar? Could this also be inherited Ar from a xenocrystic component? More robust justification is required.

Is ^{39}Ar recoil evident in some age spectra? Several age spectra have a sudden reduction in date in the higher-T steps, followed by a subsequent increase in phyllite 3779553. Is this a recoil effect, which would not be surprising given the extremely fine grained nature of the groundmass? In this case, could the older dates (e.g. 709 Ma for sample 3779554) be artificially too old? Or, is a younger, more retentive phase being degassed? This should be addressed.

I am not convinced that the ^{40}Ar/^{39}Ar date of 511±2 Ma (3779554) accurately records the timing of cleavage formation. Before making such a statement, the authors should demonstrate that this single heating step did not release Ar from any secondary alteration phases. This sample also hosts microcline, calcite and albite. E.g. did this step liberate any ^{37}Ar from the calcite, and what was its influence on the ^{40}Ar/^{39}Ar date? Does this step contains and K released from the microcline? What is the relationship between the microcline and the foliation?

Lines 314 – 324. Statements about the tectonic setting during deposition can only be made if it can be confidently established that the oldest $^{40}Ar/^{39}Ar$ dates are accurate measurements of the age of the muscovite grains. This has not been demonstrated for all of the analysed samples. This process will always be best with U-Pb concordia ages, but it is less clear when $^{40}Ar/^{39}Ar$ (single step dates!) are used. I have no issue with the logic in this paragraph, although I suggest the authors at least acknowledge this, or make a more detailed comparison between any detrital zircon U-Pb data, and their oldest $^{40}Ar/^{39}Ar$ step dates. For example, did the Mesoproterozoic grains undergo any secondary alteration before they were deposited and captured in the Delamerian Orogeny? These statements should be addressed prior to making tectonic interpretations of single step-dates.

Lines 337 – 359. The authors account for their Ar data by combining i) the time of growth/deformation, and ii) Ar loss by diffusion, which is subsequently related to exhumation. Given the mineralogy of the rocks (and the Hylogger analyses), the authors should also address the possibility of a reduction in date relative to the timing or deformation caused by fluid flow events that post-date Delamerian deformation. Could alteration be responsible for the reduction in date? If not, then why not, and use the available data to show this. The authors attach significance to the younger step-dates in sample 3779552. This sample hosts chlorite and albite, with a very low amount of muscovite. Could these younger dates simply be a result of secondary alteration? This should at least be addressed and a more robust justification is required to interpret the dates in terms of t-T paths, exhumation, fault reactivation.