

Geochronology Discuss., author comment AC3  
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## Reply on RC2

Anthony Reid et al.

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Author comment on "Complex  $^{40}\text{Ar}/^{39}\text{Ar}$  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-AC3>, 2022

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### Referee Comments 2 – Jörg Pfänder

Metamorphic rocks are an integral product of a series of complex atomic to orogen scale processes that operate over geological times sequentially and parallel at variable but mostly unknown physico-chemical and thermodynamic conditions. Deciphering the timing of these processes (i.e. discrete and geologically significant age signals) from them is a highly challenging task in geochronology. The manuscript by its title intends to contribute to this issue and takes efforts to demonstrate that step heating of low-grade metamorphic rocks (and in part mineral fractions) can provide valuable information on the age of detrital components as well as on the timing of the low-grade metamorphic processes.

Unfortunately, this goal is not reached and the manuscript does not provide convincing evidence that the detrital and metamorphic age signals can be reliably resolved.

Firstly, we thank the reviewer for their time and energy.

However, we find the sentiment of these statements surprising.

The fact that the rock depositional age is known from previous work and that some of the  $^{40}\text{Ar}/^{39}\text{Ar}$  ages in our age spectra are older than this means that they must be derived from detrital minerals in these low metamorphic grade rocks. Alternatively, the older ages are a result of excess  $^{40}\text{Ar}$ . However, the York Plots and data tables themselves demonstrate that the main age patterns are from heating steps with little or minimal excess  $^{40}\text{Ar}$ . The simplest interpretation is that these age spectra represent mixing between a detrital component and a metamorphic component, albeit that there is mixing between these throughout.

Although the data itself seems to be sound, there is a fundamental problem with the chosen methodical approach, which strictly speaking is backward-looking in that it analyzes (mostly) whole-rocks instead of carefully selected mineral separates by step-heating alone.

The point of whole rock analysis is that constraints on the tectonics/alteration/regional geology can be obtained even in situations like ours in which the rocks are too fine grained to be able to provide mineral separates. An alternative would be to undertake ultra-fine grained mineral separations such as by centrifuge, however this is not our preferred

method. The study deliberately attempted to date phyllitic rocks and other low metamorphic grade rocks to determine what results could be reliably determined from them. We believe this is a useful contribution, even should the reviewer consider this "backward-looking".

This approach has been chosen to circumvent the problem of recoil Ar loss during irradiation, which in principle is reasonable, but even pure mineral fractions having a well defined grain size spectrum often provide 'complex' age spectra during multi-grain step heating (even for single grain step heating), which results from frequently present excess argon and argon loss, controlled by thermal overprinting, recrystallisation, fluid-inclusions and/or intra-grain deformation. Therefore, without additional constraints, such as ages from other geo-/thermochronometers obtained on the same samples, or other Ar-Ar approaches such as single grain total fusion, single and multi grain step heating or in-situ dating, the gained age information remains unconstrained. This drawback has been already stated by Forster & Lister (2004) who introduced the chosen methodical approach: 'Frequently measured ages (FMAs) in individual datasets can then be recognized using statistical analysis. The significance of FMAs must be independently assessed'. This statement emphasizes the importance of doing statistics on a significantly large dataset, and the importance of independent evidence. None of both has been sufficiently well addressed in the study.

In an ideal world we would collect and analyse many more samples. However, the sample set covers a region of interest in the Delamerain Orogen, and therefore provides a significant improvement to the geochronology in this region (ie prior to this study there was effectively no  $^{40}\text{Ar}/^{39}\text{Ar}$  analyses in this region). The alternative methods suggested by the reviewer would mostly not provide more information, in fact would lose information, for example single grain total fusion or in-situ dating where information would be mixed and would not provide the information the review is suggesting.

The manuscript is also not in a shape as is expected for an international journal regarding the overall structure and writing. This, for example, refers to the organization of the samples and results chapter (sample by sample descriptions), but also to data documentation and presentation, which are insufficient .

We are uncertain why sample-by-sample treatment of data means the paper is unsuitable for an international journal. How would the reviewer suggest we approach describing the results? It seems a fairly reasonable thing to do. Many papers adopt this type of results section and enables the details of each age spectra to be brought to light and discussed. As for this not being suitable for an international journal, we fail to see the logic behind this statement.

The data further miss a thorough and careful in-depth evaluation (inverse isochron ages and intercepts of sub-datasets used to derive ages, evaluation of contributions from Ca-, K- and Cl-derived Ar-isotopes), which in particular is required given the uncommon approach of doing step-heating experiments on whole rock low-grade metamorphic rocks that contain high amounts of chlorite. Such complex samples require a more critical and sophisticated data evaluation and discussion.

The discussion is weak, not very well written, and far away from being acceptable for an international high-quality journal. It mostly remains vague, is often hard to follow, not to the point, full of unsupported and confusing statements, and contains passages that are rather empty phrases than robust scientific arguments (example: 'Complex age spectra and multiple gas domains are present in our samples from the low metamorphic grade rocks in the Delamerian Orogen. The presence of upper age asymptotes that also have

distinct isotopic compositions, is consistent with those ages being from distinct gas reservoirs. Taken together, the complex age spectra in this study clearly represent the mixing between older detrital components and younger age components ).

We will take this as a subjective comment more than a review suggestion. It is difficult to know exactly what the reviewer has in mind with these type of review comments. This type of review comment seems rather over the top, to be honest. We fail to see the logic that the paper is below standard for an international journal. The standard scientific format for a paper has been applied. Of course, we certainly aim to improve the paper and do not imagine it is ready to be published in current form. That is entirely the point of submitting a paper to the review process. Of course, it is the Editor's decision as to if our contribution meets journal standards.

We note that all the corrections for and information on isotope interferences are done and are reported on in our Supplementary Data.

The study hampers another fundamental but rather common problem, namely that the geologists among us often desire to have age information from rocks that these rocks and the available methods can not provide in an easy, fast, cheap and simple way due to fundamental physical limitations. Working on such (mineralogically) complex rocks with such a complex history (by far too many degrees of freedom) requires tremendous methodical efforts and an in-depth evaluation of the gained data by considering physico-chemical principles. Just plotting 'easy to read' age spectra plots and deriving obvious age information is not sufficient .

And yet, if we waited until we had a 'perfect' analytical data set, our results would never be published. This manuscript is an attempt to interpret step heating experiments on a series of deformed rocks. It is not intended to be the most exhaustive study of the mineralogy, chemistry, fluid inclusions and so on that could be done.

More importantly however, our main point is that complex age spectra do have meaning and can be interpreted using the concept of asymptotes and limits discussed by Forster and Lister (2009 and other publications). Necessarily the results here have been given interpretations. We fail to understand why deriving 'obvious age information' is not a valuable pursuit, especially in the context of trying to understand the metamorphic/deformation history of an area.

Nevertheless, we suspect that the reviewer is trying to tell us to dampen our 'enthusiasm' for some of the interpretations. More qualifying statements are required acknowledging the reconnaissance nature of the data. In addition, some of the samples could well be suffering due to later alteration and this is something we will look at more closely in the revision.

In summary, the manuscript needs substantial and very comprehensive revisions. A more comprehensive and systematic evaluation and presentation of all data as well as a clear and concise discussion of the results is required, along with a much better consideration of existing (methodical) literature. Doing so, and comparing the results with existing literature data, there might be a valuable scientific outcome with some importance for the community working on the evolution of such rocks and the controlling geological processes.

We will incorporate some of the methodological literature, noting the excellent, specific examples given to us by Reviewer 1.

## Chapter related comments

The abstract is in parts too vague. For example, low grade rocks also contain detrital minerals that have been overgrown during metamorphism. Then it becomes not clear that whole-rocks have been analyzed, as likely most readers will assume that a common approach has been applied where mineral separation precedes dating.

This can be rectified with a comment on the nature of the samples analysed.

Complex age spectra are interpreted as consisting of several age components. This is misleading, as it implies that discrete and real age events are present. An 'age component', however, might simply result from mixing of gas fractions from different (disturbed) grains with different ages, size and/or Ar release properties, and thus is not an age but an artifact. Would suggest to term them 'virtual age components'. This then might question the subsequent interpretation, where neither the problem of mixing of different gas fractions, nor the potential presence of excess argon or argon loss during reheating or recrystallization have been obviously addressed. This needs to be done.

The reviewer is pointing out the complexity of the age information in the age spectra, which is something we have also tried to grapple with. Clearly, we have not succeeded in providing a detailed enough description and we will work to make things clearer. The idea of 'virtual age components' is not a term we have seen used before. We will consider this further in our revision.

Line 12. 'Low metamorphic grade rocks' need to be 'Low grade metamorphic rocks' .

Why?

If there is an Editorial preference for one or the other, we would be pleased to use whichever designation is required.

The introduction is too short and misses a brief review of methods and case studies that have addressed dating of low-grade metamorphic rocks. Such a brief review should follow the first paragraph, which ends with a statement that it is difficult to separate different age signals. This is true, but several approaches likely exist and these should be briefly mentioned (K-Ar dating of shales, clay- and siltstones, etc.; Ar-Ar dating approaches such as in-situ, single-grain total fusion, etc.).

We can expand upon some of the references we cited as previous examples of work on low metamorphic grade rocks.

Line 29-30. Sentence does not make sense.

This is where responding to a review becomes quite tricky, as the authors are unfortunately left to guess the intention of the reviewer. It would be lovely to hear from the reviewer in what way this sentence does not make sense. To our way of thinking this is a fairly reasonable statement that low metamorphic grade rocks occur within orogens as much as high metamorphic grade rocks. We will nevertheless attempt to rephrase the sentence in the revision.

Line 29-30. This sentence seems somehow to end strangely.

See response to previous comment.

Line 40. ... provide information on the age of possible ....

Thank you. We will change the sentence to this.

Line 43. What do you mean by in-situ laser microprobe methods? Laser ablation in-situ Ar-Ar dating? Then this should be outlined more clearly.

Yes, this is what we mean. We will add clarification.

Line 46. 'Age populations' seems being misleading. Such commonly result from a large number of samples with a single age, or from single-grain total fusion experiments on a single or of multiple samples, and I doubt that sufficient ages can be derived from five whole-rock samples by a step heating approach to resolve statistically significant age populations.

And yet, these are the data we have available. In our revision we will provide a cumulative probability plot of all of the ages obtained in the study to clarify the more significant age populations across the samples analysed.

The geological setting chapter should be shortened by at least 15-30% , it seems a bit too comprehensive given that the goal of the paper is mostly a methodical one. Reduce the text to what is needed to understand where the samples are from and how they evolved. Rock types should be mentioned from time to time instead of only unit names (line 40-41).

The geological setting can be reduced.

Line 66. Is there a word missing after metamorphism?

Yes, "at".

Line 73. Do you mean a thermal laser? I.e. laser induced step heating ?

Yes.

The sample descriptions chapter is not straightforward to read, as it is organized in form of a sample by sample description (and in part contains useless information: e.g. line 130: 14 km east-north-east of Adelaide). The chapter should be condensed by summarizing the major features present in all samples (including the constituting minerals as these are mostly the same, just note if some untypical features/minerals are present) and by outsourcing information in a table (as in part has been already done in Table 1, why describing places in the text when coordinates are given?). The images in Fig. 4 are not very helpful, thin-section images would be much better. Either replace the field images by thin-section images, or complement them by the latter.

We can reduce the descriptive parts of the section. However, because we have a relatively small data set, we prefer to discuss the results sample by sample. This is because there are important differences between each sample that merit explicit consideration. Thin section images are worse than the field images. The samples are very fine grained.

Line 148-149. Very near: How near?

Within 15m. We will clarify this.

As the sample description chapter, the Ar-Ar results chapter describes the data and/or plots in a quite monotonous manner sample by sample. As said already, this is rather

annoying to read. The authors should reorganize it, and instead should work out the general features and describe them once, and then, supported by examples, should describe the important and significant aspects of the data.

In our view, geochronology papers should have a section that simply describes the results, in this case sample by sample. Any section that then describes general features is something that should be tackled in the Discussion, which we have attempted to do.

All ages that were derived from the age spectrum plots and which are assumed to be geologically meaningful need to be reported in a table along with all associated data (errors,  $^{40}\text{Ar}/^{36}\text{Ar}$  intercepts, MSWD, etc.; see Schaen et al., 2020; Renne et al., 2009).

We will expand Table 2 to add some of the statistical metrics for the interpreted ages. Schaen et al 2020 and Renne et al 2009 have been used as a method to report our supplementary data. All recommendations have been adhered to where applicable.

The term 'York plot' is fully uncommon. Either use inverse isochron plot or isotope ratio plot, as common in the Ar-Ar community. Ironically, the original York publications in 1968/1969 deal with linear best fit isochrons and correlated errors, a procedure that is unfortunately not applied in this manuscript, which needs to be done in a revised version.

We can re-label the York Plots as isotope ratio plot, as per the suggestion. We are aware of the issue the reviewer refers to and could go into great detail on this, however, in terms of isochrons, these are not required in this study because we recognise that we are dealing with mixtures of minerals and there is no expectation that any of the results will lie on a single isochron.

Use the term 'virtual age' instead of 'age', as it remains finally unclear whether the derived 'ages' are of geological significance or represent averages of different events with different ages.

Does the reviewer mean 'apparent age'? We have not come across the idea of a 'virtual age'. We will consider this in the revision.

Line 196-199. What do you mean by a more or a less retentive domain? How does the physicochemical process of retention (i.e. diffusion) affect an isotope ratio? Not significantly to my opinion if we ignore diffusive isotope fractionation. You may tap different domains with different isotope abundances within a mineral, or different minerals at different temperatures, but just using the term 'retention' is insufficient to cover the complexity of degassing a multi-phase system.

We will remove 'retentive' from the sentence.

Line 201. The term asymptote needs to be defined. What is the 'concept of asymptotes' as mentioned already in the abstract (line 17-18)? What you mean is simply an upper or lower age limit. So the term asymptote is misleading here as in a strict mathematical sense an asymptote is a limit to which a variable converges.

We will remove the term asymptote and instead refer to limits.

Line 209. What do you mean by the statement ' $^{36}\text{Ar}/^{40}\text{Ar}$  values have no atmospheric component'? This sentence is fully meaningless, if not wrong. Any gas fraction analyzed during a step heating experiment that provides detectable  $^{36}\text{Ar}$  potentially can contain a fraction of atmospheric argon (i.e. an atmospheric component). In principle, any datapoint in an inverse isochron diagram that does not plot on the x- or y-axis can be interpreted as representing a three-component gas mixture between atmospheric argon (plots on the y-

axis at 0.0033), radiogenic argon (plots on the x-axis at its 'true' age  $^{39}\text{Ar}/^{40}\text{Ar}$ ) and a third component ('initial' Ar) that plots on any point on the y-axis except at 0.0033. Data scatter then simply reflects changing mixing proportions between these three 'components' during step heating.

The heating steps we are referring do indeed plot on or very near the x-axis. However, the statement should read "virtually no atmospheric component" or perhaps "very minor atmospheric component". Regardless, the statement can be omitted in the revision and not change the interpretations.

Line 212. What do you mean by 'two domains'? Does not make sense to me.

Domains in the sense of gas reservoirs within the composite mineral aggregates. An older component of gas, say from detrital minerals within the whole rock is one 'domain' of gas age, compared to a newly grown 'domain' of gas with a younger age. This can easily be clarified in the revision, with the word 'domain' being changed to something less specific.

The Discussion is not appropriate to an international journal. There is no but some vague geological outcome from this discussion. Although the overall approach of this study (chloritized whole-rocks) in principle is questionable, it might be worth doing such an investigation in an effort to circumvent the problem of recoil loss of  $^{39}\text{Ar}$  in fine grained mineral separates during irradiation. The way it has been done here, however, is insufficient as the data have not been evaluated in depth.

We have done our best to write this for an international journal. We believe that the geological meaning we can extract from the results are in fact all the more remarkable because we have sampled chloritized rocks. We will do our best to make the revision better.

Line 239-242. Why just different mineralogy? Mixing of the same mineral type with different ages will also provide complex age spectra. Please clarify.

Agree.

Line 243. The term crystallization in this context is misleading. A low grade metamorphic rock can't be described by a discrete 'crystallization age'. Be more clear what the maximum could be (an old detrital component).

Yes, this can be clarified.

Line 244-245. What is a geological resetting event in this context? Be more clear. And note that a 'less retentive domain' is a physical representation of a thermal 'resetting' event. So using 'or' in this context is misleading.

We will reword this statement. A resetting event could be any type of process that resets the argon age systematics within a sample. The least retentive domain is just that. It is the domain that has closed to argon diffusion last in the cooling history of a mineral or is the most easily reset by any process that causes resetting of the argon clock within that domain.

Line 254-255. What is the 'main mineral gas reservoir'? What you mean is the other endmember, the purely radiogenic component (see above). Be more clear. Why should this mixing be the result of 'deformation-induced recrystallization'? This is a fully wrong statement, as this mixing is simply due to different argon 'components' having different diffusivities (or retentivities), or different bonding energies, or whatever we want to term it. The presence of atmospheric argon and radiogenic argon has not necessarily to do with

'deformation'. The latter may trigger partial argon loss (and also uptake of argon), but this is another issue. In an inverse isochron plot, any Ar loss and any excess component force the data deviating from plotting along a straight line.

Yes this can be clarified.

Line 260. The statement 'The presence of upper age asymptotes that also have distinct isotopic compositions' is circular. The whole sentence is not understandable.

Will revise the sentence. The intention is to highlight that the older age components are distinct from younger ages in terms of their age as well as their composition (K/Ca etc).

Line 285-287. Hard to understand what is meant here.

Will attempt to revise.

Line 289. State from where the deposition age is known .

The age of deposition is given in the Geological Setting (and in Table 1), which the reviewer wishes us to reduce in length. We will try to incorporate some of these details into the Discussion where required.

Line 292. What are similar ages? How similar? Provide data please.

This can be clarified.

Line 303. What is the depositional age and from where is it known?

The age of deposition is given in the Geological Setting (and in Table 1), which the reviewer wishes us to reduce in length. We will try to incorporate some of these details into the Discussion to clarify.

Line 317-324. These setting inferences from very questionable 'ages' derived solely from age spectra plots obtained on very complex samples are very daring.

If required, this section can be removed from the Discussion. It is not central to the main point of the paper.

Line 353-354. Or simply a stronger resetting of smaller grains. I do not like the term 'retentivity domains'. It makes things mystic in some way, and obscures that we were dealing with a complex mixture of crystalline material. What do we physically have? We have rock fragments. Each fragment consists of a number of different mineral types, where each type has its own grain size distribution (affecting diffusivity, or retentivity). Each mineral, in the simplest case, may have its own age, but may be compromised by either Ar loss or the uptake of excess argon (or both), possibly at different times (multiple events). Clearly, this complex material and history will provide complex age spectra, but interpreting them solely by considering 'mystic' domains does definitely not do justice to the complexity of the problem. Therefore, data interpretation needs to be done in the light of what is physically present.

Yes, the resetting can be affected by grain size. We will modify the sentence accordingly.

Line 349-350. This is completely at odds. If 'ages' out of a simple sample are younger than other ages from other samples, how can this be indicative for 'a complex history of mica growth and cooling'? Such a statement is fully unsupported.



Can reword this.

Line 358-359. Useless statement.

Can delete this.

Line 372. ... low metamorphic grade ...

Yes.

Tables

Table 1 . First line: 515 Ma: Provide the exact datum including error (line 149) as in the second line.

Yes.

Table 2 provides too little information. The power of Ar-Ar is the fact that a dataset provides internal control on consistency. Just reporting ages and errors is insufficient.

More detail on the type of age and statistics will be added to Table 2.

Figures and figure captions

Fig. 1 is a good overview. Needs to be 'Damara Zambesi Orogen' not 'Darmara Zambesi ...'

Yes.

Fig. 2. (a) and (b) are missing. Make it more clear that the red square in the lower left sketch outlines the region shown in (a) and (b). Enlarge the sample location dots.

Yes.

Fig. 3 should be deleted. It provides nothing necessary to support the goal of this study, and contains information that in part can be found in the text (and is mostly speculative). Three large images with geological maps and profiles in a paper with a methodical goal are too much.

The figure is intended to help the reader visualise the orogen and relate the low metamorphic grade sections to the eastern higher metamorphic grade regions. However, the figure can be deleted if required.

Fig. 5. The way the data is presented is mostly insufficient. First, the y-axis scale in some of the age spectra plots is too large, making it difficult to resolve individual steps and differences between them. Adopt this even at the expense of having the same scale in any of these plots.

We prefer to leave each in a standard view as this enables easy comparison between the age spectra. However, as Reviewer 1 and 2 have both asked for this, we will replot the age spectra.

In addition, provide in the figure caption or in the figures what initial  $^{40}\text{Ar}/^{36}\text{Ar}$  has been used to correct the isotope composition of each step for initial argon in the procedure of calculating individual ages for the age spectrum plots (I guess that an atmospheric value has been used, but which one?).

Will be added to the caption.

Second, if an age is resolved from an age spectrum, an inverse isochron through the respective steps needs to be plotted along with the corresponding inverse isochron age and inverse y-axis intercept value (i.e. the  $^{40}\text{Ar}/^{36}\text{Ar}$  of the initial argon). If the latter deviates from the atmospheric value, this needs to be considered in age spectrum age-value calculation and the potential causes need to be considered (except in cases where the correction can be neglected due to near-100%  $^{40}\text{Ar}^*$ -values).

This approach is an interesting one and would be valuable were these analyses of single minerals. In this study, the maximum and minimum ages defined are often defined by single steps, or a small number of steps. As a result, it is impossible to calculate an isochron age. If the results were averages of multiple steps, working out an isochron age from those data points would be a good idea. We will however, carefully go through our 'ages' and determine if this approach is appropriate.

Denote which steps in an age spectrum have been used to calculate which virtual age. And mark the temperature steps with numbers in both the age spectra as well as the inverse isochron plots so that one can see which step belongs to which in each plot. Mark the position of each of the derived 'ages' from the age spectrum at its corresponding x-axis position in the inverse isochron plot.

The York Plots can be replotted with the step numbers.

And include the datapoint errors in the inverse isochron plots.

The York Plots can be replotted with the data point errors, however, in most cases the error is smaller than the symbol.

Most of the upper 'age values' in the age spectra plots are hardly of any geological significance. For example, in Fig. 5c the age of  $371.1 \pm 7.8$  Ma. From which steps has it been derived? There is hardly anything that looks like a plateau. Do you really believe this age is of any geological meaning? Hard to believe, the steps are more or less continuously increasing. And as far as I can see, hardly any (sub-)dataset in the inverse isochron plots will provide an atmospheric intercept. Something as for example in Fig. 5d needs to be interpreted, to my opinion the plot suggests Ar-loss for the very low temperature steps, and lots of excess Ar in the steps that deviate to the lower left side (same for Fig. 5h).

We will rephrase the interpretation of this spectrum. No we don't think the younger age has any meaning, and we attempt to clarify this in the paragraph on line 348. We note that there is no reason why we should expect anything that looks like a plateau in a mixture.

Fig. 6. The whole explanation is insufficient and not comprehensible. What, for example, is meant by 'Model age spectrum of a simple age spectrum with two component system, mixing between more and less retentive argon gas reservoir.?'

The reviewer would find answer to this in the original paper from where we reference this model age spectra. It is a model in that it is not real data, it is a hypothetical situation where an age spectra is obtained from degassing a two component system with a highly retentive domain and a less retentive domain in a step heating experiment. The original paper is Forster and Lister (2004). We will provide more detail to this in the revision.

Is the reviewer referring to the figure caption in this statement of it being 'insufficient and not comprehensible' or to the discussion in paragraphs on 238? If the former, we will try

to flesh out the figure caption, however, aside from the caption we have provided, we are unsure what is being asked of us in this comment.