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Interactive comment on "Precambrian faulting episodes and insights into the tectonothermal history of North Australia: Microstructural evidence and K–Ar, ^{40}Ar–^{39}Ar, and Rb–Sr dating of syntectonic illite from the intracratonic Millungera Basin" *by* I. Tonguç Uysal et al.

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General comments Uysal et al present a large dataset of geochronological, mineralogical and petrographic data from fault rocks and host rocks in the Millungera Basin in North-Western Queens- land, Australia. The geological history of the basin is largely unknown and therefore the data represent a potentially good contribution on the geological history of the area. The text is generally clear and well written with some occurrences of imprecise or unclear terminology, which I attempted to address case by case, see the line numbered comments below. Although the data appears to be generally of good quality, there are items missing which prevent detailed evaluation or further use of the data – IE a data table for 40Ar/39Ar geochronology (in the appendix is OK) stating irradiation time / J values and signal volts for the different isotopes; the used decay constants in all isotopic methods, the uncertainties in the 87Rb/86Sr ratios (necessary for calculating isochrons), We cannot present 40Ar/39Ar irradiation times/ J values and signal volts, since this analysis was done at the Michigan University by Chris Hall who has already retired since then. Chris provided us only data in Fig. 6, and in many journal papers published by the Michigan group, only similar data set has been used. Therefore, we have not requested times/ J values and signal volts data additionally. We mentioned in the text that we used decay constants recommended Steiger and Jäger, (1977). For Rb-Sr, we used one that was recommended by Villa et al. (2015). For isochron age calculation, standard errors of $\pm 0.01\%$ for 87Sr/86Sr and of $\pm 1\%$ for 87Rb/86Sr ratios were assigned to the results. Individual analytical uncertainties were generally smaller than these values (please see section 3.3 in the revised manuscript). and the uncertainties for 40Ar* and K2O determinations for K-Ar. Further- more, in text, sometimes errors are discussed as 2σ, sometimes as 1σ for a single method (IE K-Ar, further discussion in the line numbered comments below). We noted the relevant revisions in the line numbered comments below). Three different dating methods (Rb/Sr isochrons, K-Ar and encapsulated 40Ar/39Ar) have been used to extract age information from numerous clay samples, which may or may not be mixtures of different generations of illite. In the present dataset, when various illite types are identified by XRD, their presence in different proportions appears to yield similar K-Ar ages, which suggests that the K-Ar isotope system was closed (IE by dropping temperature) or started (due to neo-crystallization) at the date recorded by the samples. Unfortunately illite polytype data are not provided for most of the samples, presumably because it requires a substantial sample amount compared to K-Ar analysis. Conceivably the Kübler and Arkai indexes can provide some insight but these data are not significantly discussed in text. It is not likely that 2M illite is formed in the "diagenetic" zone according to the Kübler index, for example. It is correct that illite polytype data are not provided for most of the samples, because it requires a substantial sample amount compared to K-Ar analysis, which could not be extracted from core samples with a limited size. We created a new section in the revised manuscript for a discussion on the Kübler Index data and their correlation with K-Ar ages (please see 5.4. Changes of illite crystallinity in relation to K–Ar ages). We also discussed in this section that that 2M illite can coexist with illites from the "diagenetic" zone. Generally, it seems like the host rock has a similar illite age as the fault gouges. Then the interpretation of the illite ages from fault gouges, as ages of faulting needs an expanded discussion. Is it not possible that the faults were formed in a first stage, and that both fault and host rock experienced fluid flow and illite growth at a later stage (IE 1100-900 Ma)? In the second part of section of " 5.1. Faulting, fluid-rock interactions and clay generation", we discussed that faulting and regional fluid flow occur in association. Large volumes of fluids are expelled as a result of faulting, leading to the generation of hydrothermal/geothermal systems affecting the surrounding host rocks. We are not saying that both fault and host rock experienced fluid flow and illite growth at a later stage. Later illite growth with younger K-Ar ages are results of younger faulting and/or thermal-fluid flow events. This has been discussed in the in the revised manuscript (5.3. Changes of illite crystallinity in relation to K–Ar ages). Also, one of the existing hypotheses is that this basin is Paleozoic- Mesozoic based on regional considerations (Korch et al 2011). Thus, if the illite is authigenic, then the Mesoproterozoic age of the basin is a major conclusion, and its implications should be further explored. We discussed this issue in the revised paper in section 5.5: "The age data from the fault gouges provide clear evidence for a late Mesoproterozoic minimum age for the Millungera Basin, and is in accordance with the early-mid Mesoproterozoic maximum depositional age of the Millungera Basin as constrained from zircon ages for Millungera Basin sandstones (Neumann and Kositcin, 2011)." In my opinion therefore, the dataset could vastly benefit from more detailed, well structured discussions on what is actually being dated, both from a mineralogical

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and macroscopic point of view. A section evaluating age vs. polytypes, when available, and age vs. crystallinity is currently lacking. A section (5.4 in the revised paper) with a discussion and figures 10 and 11 (in the revised ms) evaluating age vs. crystallinity has been created. _________________________ Method specific comments 40Ar/39Ar with 39Ar encapsulation for recoil correction: An implicit assumption of 39Ar recoil measurements is that the sensitivity of the measurement of 39Ar is the same as during the measurement of the rest of the sample, because we are measuring volts as an independent variable during the 39Ar release from the vial and combining them with voltage measurements during step heating, where normally only voltage ratios are used (which is typical for the 40Ar/39Ar method. However, in most mass spectrometers, ionization efficiency is a function of total gas pressure in the ion source. For example, Burnard and Farley (2000) show that 40Ar* sensitivity increases by 20% when increasing the argon pressure by a factor of âĹij50 on a MAP mass spectrometer. Normally 40Ar/39Ar irradiation durations are planned to have a 40Ar*/39Ar ratio of 100-300. It follows that the gas pressures during 39Ar recoil measurements may be different by a factor of >100 from the gas pressures during step heating, and therefore it is not permissible to do recoil corrections simply by adding volts together, without tuning the sensitivity in the expected pressure ranges and correcting the volts for this calibration before calculating the total gas age. This problem may be exacerbated for old samples with lots of 40Ar*, particularly if irradiation times are chosen for a rather conservative J value. More generally, have illite ages this old ever been calibrated between K-Ar and 39Ar recoil corrected 40Ar/39Ar analyses? I am not sure, which leads to the more general question – are recoil-corrected 40Ar/39Ar ages really equivalent to K-Ar ages? Perhaps the attempt to base geological constraints on the 40Ar/39Ar ages is not sufficiently warranted. Since we have only 4 Ar-Ar ages in this study, reviewer's comment would be useful for a future method paper to compare K-Ar and Ar-Ar ages. In our case, illite ages this old have not been calibrated between K-Ar and 39Ar recoil corrected 40Ar/39Ar analyses. However, according to Clauer et al. (2012, Earth-Science Reviews 115, 76–96) and Clauer (2020 personal communication), Ar-Ar total gas ages of encapsulated samples should be comparable to K-Ar ages (although they report some differences as well). Indeed, we obtained identical Ar-Ar, K-Ar and Rb-Sr isochron ages for fault gouge and matrix illites from a number of geological settings in Australia (e.g., Middleton et al., 2014; Rosenbaum et al., 2015; Babaahmedi et al., 2019; Uysal et al., in prep.). Method comments Rb/Sr In the data tables 1 and 3, it should be clearly marked which samples were used for which isochron age calculation. Furthermore, the error of the 87Rb/86Sr ratio should be specified as it is required to reproduce isochron calculations, and the referenced constants used to calculate the ages In Table 3, it has been marked which samples were used for which isochron age calculation. In line 513 you mention discrepancy between the Rb/Sr and K-Ar ages are possibly due to heterogeneous samples. Are you suggesting that the sample homogenization before splitting between Rb/Sr and K-Ar was not effective? Or do you mean something else by heterogeneity at the micro scale? Homogeneous sample material is a prerequisite for K-Ar geochronology. Clay size fractions have millions of particles in a typical argon aliquot size. If the samples were adequately homogenized during preparation, within size-fraction heterogeneity seems like an unlikely problem. Alternative suggestion - as is documented by XRD with the crystallinity, the polytypes (when available) and the K-Ar ages of these fractions, there may be different generations of illite. In section "4.1 Sample description and micro structures", we clearly presented that injection of cataclastic, hematite and clay-rich layers are common, which we relate to multiple slip events. Therefore, the sample homogenization before clay separation and splitting between Rb/Sr and K-Ar was not effective for some samples. For example, multiple injection layers in samples Dob-441, Dob-449.1 and Dob-449.3 are obvious even in macroscopic scale (Figs. 2, 4, 5). Analysing various size fractions helped to minimise the effect of multiple events to create mixed ages. Additionally, in the revised paper, we evaluated K-Ar ages in relation to Kübler Index (KI) values and grain sizes, whereby consistent K-Ar ages of samples with different KI values and grain sizes (Fig. 7b, and Fig. 11 in the revised paper) are interpreted as geologically meaningful. It is possible that the Rb/Sr "isochrons" are mixing lines between older and younger components. A common test for mixing is to plot the 87Sr/86Sr ratios vs. 1/(total Sr). Colinearity suggests mixing (e.g. Faure 1977, Zheng 1989, Chemical Geology). In the case of these samples, there is a clear correlation between 87/86 for most samples. Is it not plausible, then, that the "isochrons" you plot are in fact mixing lines instead? A discussion on this possibility is lacking from the current manuscript but is necessary in evaluating isochron data. If, as the authors suggest, the Rb/Sr system is more resistant to thermal resetting than the K-Ar system, is it possible that the K-Ar ages are variably reset near 1 Ga, whereas the Rb/Sr systematics display two component mixing? Together, it seems that the data suggest several illite generations, which suggests that the components in the samples are not co-genetic, but rather mixtures, and thus this very real possibility that the isochrons are instead mixing lines, should be discussed. We created a new discussion dealing with the above comments (section "5.2. Geochronology: comparison between Rb–Sr, K–Ar and 40Ar–39Ar ages" in the revised paper). It is a well-know-fact that the partial melting or the presence of various components with different 87Sr/86Sr initial ratio in a magmatic body causes mixing line rather than an isochron, which is common in high temperature environments. However, as we stated in the revised section 5.2: "valid and geologically significant isochrons and mixing lines can also be obtained simultaneously from samples with different mineral populations, comprising minerals with different Rb/Sr ratios but identical initial 87Sr/86Sr ratios. In this case of identical initial Sr-isotopic compositions of two components of a mixture at time t=0, the two components and mixtures thereof define a horizontal line both in a classic isochron diagram (which is the key condition for validity of calculated Rb–Sr isochron ages), and in the 87Sr/86Sr vs. 1/86Sr diagram commonly used for evaluation of binary isotopic and compositional mixing (cf. Wendt, 1993 and Schneider et al. 2003 for theoretical background)". See the revised section 5.2 for further text on this topic. We recently discussed about this issue with Norbert Clauer and Johannes Glodny, who have also been working on Rb-Sr systematics for low temperature and metamorphic environments for many years. ________________________ Line numbered comments Line 43 – "cores from borehole shaft or tunnel sites" – do you mean bore-

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hole shafts? Or boreholes, shafts or tunnel sites? Shaft deleted Line 49 – “reveal” is maybe better than “define”. Agree, done. Thank you. Line 60 – do you mean previous studies in north-central Australia as suggested by the flow of the text? Or worldwide? It is clearly mentioned in the text that we deal particularly with north-central Australia. Line 73 – is the potential reactivation history relevant to the discussion? This general statement may not be necessary. Also, Viola 2013 is missing from the reference list. Fixed. Thank you. Line 131- relative errors of each of the 1M and 2M polytypes? Or the relative error on the %2M1/total illite? Or the absolute error? Absolute, fixed. Line 173 – 1 or 2 σ? The error of K determination of standards is better than 1.2% 1σ (or 2.4% 2σ). This sentence has been added in the revised ms. Line 181- this is repetition of line 179 Yes, thank you. Fixed. Line 182 – The error of which air shots is 0.2??? The 16 40/36 measurements you report have a standard deviation of 1.23, and clearly these values do not overlap within error (excess scatter is common in argon geochronology). It is not clear what your quoted uncertainty represents. Changed as: “The error for the 40Ar/36Ar value of the airshot yielded 296.08 $\pm$1.23, (0.41%) 1σ.” Line 183 – you quote 2 σ uncertainties of 1% for argon measurements, yet your HDB1 analyses yield concentrations with a 1 σ standard deviation of 1.1% relative, for a material which is far more ideal than most samples. Are you sure you don’t mean 1 σ uncertainties? Is your standard deviation of spike calibrations (σ(X) in the error equation from Cox and Dalrymple) better than 0.5% RSD (which it has to be if the total uncertainty is better than 1% 2 σ)? Also, please use the same uncertainty level (1σ OR 2σ) throughout the paper. 1σ is generally fine to use for K-Ar. Changed as: “The general error for argon analyses is below 1.3% (1σ) based on the long-term precision of 330 Ar measurements of the international standards.” Figure 3 is at present too low resolution for the reader to be able to follow your de- scriptions referring to this image throughout the text. Please provide higher resolution images. Unfortunately, low resolution was a result of converting whole ms to pdf. For final submission this figure will be much better. Figure 4 – Muscovite should be abbreviated by Ms. It looks like the muscovite is oriented along the S shears, is it possible that the muscovite is authigenic with deformation? This whole section is very nice otherwise. The abbreviations were corrected both in the figure and figure caption. Line 266 – “an ultrafine grained oriented at a small angle” – it appears that a noun is missing here. Fixed, thank you:” . . .by disrupting the grains into a plane composed of ultrafine comminuted grains oriented at a small angle ($\sim 20^\circ$) to the macroscopic shear surface. . .” Line 281 – I don’t understand what you mean by “to compare their metamorphic grade with that of the fault gouges”. Agree, unclear meaning, hence deleted. Thank you. Line 292 – SEM analyses on the bulk rock? <2ïĄ■m size fractions. Included in the text. Line 296- on the right-hand side (as opposed to site). Also, this sentence is not very clear – are you implying that the detrital illite has straight edges in this study or not? In this sentence, do you mean to say, “unlike those presented in this study”? Thank you for pointing out this confusion. We fixed the sentence in the text: “A number of previous studies (e.g., Clauer and Liewig, 2013) showed that detrital illitic clay particles rarely have straight edges, but rather occur in particles with diffuse-blurred and irregular edges (Fig. 3c, like the white material on the right-hand site).” Table 1, Please mention that the quoted uncertainties are 1σ (as you say ine line 312). Also, the illite percentages are relative to total illite, not the total percentage in the bulk rock. This should be specified in the caption. Done, thank you. Table 2 – please show the reference values you use for comparison. Generally, I think it is good practice to show the uncertainties on the individual measurements (K, 40Ar*). The uncertainties on the ages are not visible here (the table is cropped). The final error on the age is shown in this table “Error Ma”; it incorporates all other errors: Error on K, error on spike calibration, etc. Line 311 – Obviously from the graphs in figure 6, F(recoil) is the fraction, not the per- centage, which would mean 5-12% recoil loss. Yes, corrected accordingly. Line 312 - “data” is a plural term, so “data are”, not “data is” Yes. Line 329 and further on in this section – you document varying amounts of 2M vs 1M in sample JC360.7. Taking a weighted mean of different ages implies that you are dating the same material, which is obviously not the case for this sample. If you suggest that the 2M and 1M were formed simultaneously this is permissible, but then this should be more clearly discussed in the discussion section and such averaging should be moved

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to the discussion section, as it is an interpretation. We took weighted mean of the identical or overlapping ages within analytical error for different size fractions from the same samples. We think that averaging of ages should not be presented in the interpretation/discussion, but rather be in the result section. We already mentioned in the introduction section that previous studies documented that simultaneously formed 1M and 2M illites can occur in fault gouges. We also discussed this in section 5.4 in the revised paper: "Although the 2M1 polytype has been known to appear at temperatures higher than 250ïĆřC (Srodon and Eberl, 1984), its occurrence at lower temperatures at about 200-250ïĆřC in co-existence with 1M/Md has also been reported (Walker and Thomson, 1990, Chen and Wang, 2007; Hejing et al., 2008)." So, co-generation of 1M and 2M illites is not uncommon at all, and seen also often particularly in hydrothermal systems bearing ore deposits (reported in many economic geology papers!). Line 333 – total gas age of fraction <2μm (specify the fraction). What does this discrepancy mean, it is present for half the 40Ar/39Ar analyses? And if you trust the K-Ar data from that sample, why do you compare the discordant 40Ar/39Ar age from one sample to K-Ar ages from other samples? Clauer et al 2012 present numerous comparisons of K-Ar vs encapsulated 40Ar/39Ar data and there often (if not to say usually) is a discrepancy. We specified the fraction and modified the text. Thank you. We have only 4 Ar-Ar ages for <2 ïĄ■m fractions, and therefore the main task of this study is not reporting the comparison of K-Ar and Ar-Ar techniques, although, as mentioned above, we reported previously a number of examples of consistent Ar-Ar, K-Ar and Rb-Sr isochron ages for fault gouge and matrix illites from various settings (e.g., Middleton et al., 2014; Rosenbaum et al., 2015; Babaahmedi et al., 2019; Uysal et al., in prep.). However, we agree with the reviewer that discordant 40Ar/39Ar age from one sample should not be compared to K-Ar ages from other samples, and we corrected this in the revised text. Line 336 – total gas age. No, >2 ïĄ■m fraction of sample JC-408 was analysed only for K-Ar, which is a total gas analysis anyway. Line 339 – Same as before. I realize it is impossible to do polytype analyses on some very small fractions, however XRD data from coarser samples suggest that ages get younger with decreasing 2M. Therefore, it is plausible, that the youngest ages (without polytype quantification) have more low-crystalline 1M illite which is younger. Yes, we agree with this comment. Although a lot of 1M-rich samples give consistent ages as the 2M-rich samples, finest size fractions with younger ages should contain younger 1M illite phases. Supplementary data – you have several XRD plots for the same samples which are not grouped together, please group them. Also, you have several analyses for the same fractions with different scale, but the graphs are different, please clarify what those graphs represent. From the comment of the reviewer it follows that he cannot see the entire supplementary data. In the supplementary file, first XRD pattern of random powder size fractions are presented, followed by the oriented clay XRD analyses for samples from each borehole from top to the bottom of pages according to their stratigraphic depths. Line 369 – 371 – this is an interpretation, move it out of the results section. Agree, this is an interpretation, which however makes sense within the course of the sentence. Line 376- the lines in figure 8a are technically "reference isochrons" as they only ap- proximate the data and are not formed by regression. It is a good idea to show this, but it should be labeled appropriately. The other graphs in this figure are appropriately labelled. Fig. 8a cannot be regarded as a reference isochron (reference to what?). Fig. 8a shows 3 different clusters of data points defining 3 parallel lines, where names of all samples are labelled, of which Rb-Sr analysis were done. These are not meant to represent isochrons. Since the conversion of the original graph to the pdf file, the resolution has decreased significantly. Line 377 – which samples? "Some samples plot between these lines (Fig. 8a)". Corrected accordingly. Figure 8 – please provide higher resolution graphs –in vector format if possible. The original figure has much higher resolution, which I cannot submit at this stage. Line 475 – This is indeed the case for micas formed during UHP metamorphism, in which case white micas recrystallize below the 40Ar* closure temperature and/or incorporate 40Ar* from the host rock; however I never heard of this being a problem in "normal" settings. Thank you for the comment. Line 508 – Although I largely agree with the discussion, I disagree that the K-Ar age of finer fractions of 922.2 ± 21.2 is consistent with the Rb/Sr age of 1000 ± 12. My own cursory reading of the (limited) data on the Millungera basin shows that the thermal gradient is extraordinarily high (60-70 degrees/km, Korsch 2011). Is it possible that high thermal gradients partially reset the illite K-Ar system at âĹij900-1000 Ma? Yes, the reviewer is right, we made a mistake, and corrected the text accordingly. On the other hand, extraordinarily high geothermal gradient (60-70 degrees/km, Korsch 2011) in the Millungera Basin is reported for recent times. Line 509 – I disagree when half of the 40Ar/39Ar ages are very different from K-Ar ages from the same fractions. Ok, we deleted 40Ar/39Ar in this statement. Line 536 – Granites with a thickness of 5 km ? do you mean, granites 5 km below the surface? Granites with a thickness of up to 5 km, changed. Section 5.4 – Korsch et al 2011 tentatively suggest that this is a Mesozoic basin. This hypothesis appears to be clearly contradicted by the data in this paper. As we also discussed in our paper in section 2 and 5.5, zircon age data constrain the maximum depositional age of the basin (Neumann and Kositcin, 2011): “The age data from the fault gouges provide clear evidence for a late Mesoproterozoic minimum age for the Millungera Basin, and are in accordance with the early-mid Mesoproterozoic maximum depositional age of the Millungera Basin as constrained from zircon ages for Millungera Basin sandstones (Neumann and Kositcin, 2011)..” See section 5.5 in the revised paper. Line 597 – no tectonic event preserved after 905 Ma – this conclusion is only true if i) you consider 1M and 2M illites to form simultaneously, or ii) if the youngest sample has no older illite. Yes, this is the case, and this has been further discussed in section 5.4 (please see also Fig. 11) in the revised paper. I hope that the authors take my comments constructively. Indeed, we thank the reviewer for his very helpful and constrictive comments.

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