

# ***Interactive comment on “Precambrian faulting episodes and insights into the tectonothermal history of North Australia: Microstructural evidence and K–Ar, <sup>40</sup>Ar–<sup>39</sup>Ar, and Rb–Sr dating of syntectonic illite from the intracratonic Millungera Basin” by I. Tonguç Uysal et al.***

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General The manuscript by Uysal et al. provides a large dataset of geochronological (K–Ar, Ar–Ar, Rb–Sr), X-ray diffraction (Kübler and Árkai indexes; illite polytype determinations) and geochemical (trace elements) data from fault and host rocks collected from two boreholes located in the Millungera Basin, Australia. Such an integrated approach aims at determining the previously unrecorded Proterozoic tectono-thermal

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events which affected the Basin. The text is clear and well written, and data are of good quality. Some parts of the text require additional background information and a more focused interpretation of the data. 1) The geological setting is not informative and more background information is required on the region. The authors should briefly describe the main orogenic stages affecting north-central Australia and adjacent basins (e.g. Georgina Basin) since the Musgrave orogeny to let readers understand the interpretation of geochronological data in the framework of regional tectonics. In the discussion section, at least five important tectono-thermal events have been linked to various orogenic phases and magmatic events but no information was given in the geological setting. Furthermore, this section would benefit from a line drawing of the seismic line 07GAIG1 or from borehole stratigraphy (at least for the collected sandstone intervals). The geological setting has been extended significantly, by adding a new section: “2.1. Regional tectonic history”, describing the main orogenic stages affecting north-central Australia and adjacent basins (e.g. Georgina Basin) since the Musgrave orogeny and before. In addition, key geological interpretations of the seismic line 07GAIG by Korsch et al (2011) were included in the text and the relevant cross section is presented as Fig. 1b. We referred to Faulkner et al. (2012) and (Fitzell et al. (2012) for a comprehensive borehole stratigraphy for Dobbyn 2 and Julia Creek 1. These are publicly available (online) reports. 2) Samples from your study have been collected from thrust faults at the margin of the Millungera Basin suggesting basin inversion during the Alice Springs orogeny (400- 350 Ma). Why K-Ar and Ar-Ar dating did not record the mid Paleozoic Alice Springs orogeny? Why don't you have neoformation of synkinematic illite during that thermo- tectonic event? May this depend on depth of deformation that may be different between thrust and extensional tectonics? Or did the absence of circulating fluids during faulting affect illite formation? A more detailed discussion on this topic is needed. This issue has been discussed in the revised manuscript. In the newly created section (5.4. Changes of illite crystallinity in relation to K–Ar ages), we discussed the temperature and fluid-controlled conditions for resetting of K-Ar isotopic system of illitic clay minerals. Additionally, we have a statement in this regard in the revised paper

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in section “5.5. Significance for regional tectonics”: The southern Burke River Fault, just to the west of the Millungera Basin (Fig. 1), represents a rift–bounding normal fault, were reactivated and inverted to reverse faults during the mid–Paleozoic Alice Springs Orogeny (~400–350 Ma) (Greene, 2010). Similarly, samples from this study were taken from thrust faults at the margin of the Milungera Basin. However, the  $^{40}\text{Ar}$ – $^{39}\text{Ar}$ , K–Ar, and Rb–Sr ages of the fault gouge illites have been essentially preserved and no tectonic event after about 905 Ma has reset the isotopic systematics of these fault gouges (Fig. 10). This can be explained by the lack of significant fluid or heat flow events allowing recrystallisation or  $^{40}\text{Ar}$  diffusion from illites. 3) Kübler (KI) and Árkai (AI) indexes have not significantly discussed in the text and a comparison of ages vs. illite polytypism or age vs Kübler and Árkai indexes is lacking. The discussion section would benefit from such a comparison. In some cases, Kübler index values indicate diagenetic conditions but most of the illites are 2M1 crystals. This is not very likely and needs a comment. Additionally, KI values from DOB borehole indicate highly variable temperature conditions from early diagenesis (KI=1.0 and T about 100°C) to epizone (KI=0.21; T>300°C) in a very short interval of depths or for different grain size fractions in the same sample (e.g" dob-389). Authors should provide a geological explanation for such difference in temperature. Alternatively, do KI data record the maximum temperature conditions during faulting or hydrothermal activity, or they provide, in this case, misleading interpretation for very low grade metamorphic zonation? Are KI data reflecting a mixing of authigenic and detrital illite/muscovite populations? It would be also useful to introduce the boundary limits of very low grade metamorphic zones (Diagenesis-anchizone, anchizone-epizone) as calculated by Kübler and Árkai indexes in the CIS scale in the “XRD and SEM clay mineral analysis” section. Now Kübler (KI) index has been significantly discussed in the revised manuscript (see 5.4. Changes of illite crystallinity in relation to K–Ar ages. See also new Figures 10–11). Since chlorite and kaolinite coexist in the majority of samples with overlapping peaks around 7Å (002 chlorite peak), Árkai (AI) index could be measured only for some samples without kaolinite. A comparison of K–Ar ages vs. illite KI indexes has been done

(see Figure 11 in the revised paper) and a detailed discussion has been presented in section “5.4. Changes of illite crystallinity in relation to K–Ar ages”.

Re – In some cases, Kübler index values indicate diagenetic conditions but most of the illites are 2M1 crystals. This is not very likely and needs a comment: The transition from 1M to 2M1 illite is gradual rather than abrupt in many geological environments. Therefore, diagenetic 1M illite can coexist with 2M1 illite. There are examples in the literature. We have this discussion in the revised paper in section 5.4: “Although the 2M1 polytype has been known to appear at temperatures higher than 250°C (Srodon and Eberl, 1984), its occurrence at lower temperatures at about 200-250°C in co-existence with 1M/Md has also been reported (Walker and Thomson, 1990, Chen and Wang, 2007; Hejing et al., 2008).”

Re – Additionally, KI values from DOB borehole indicate highly variable temperature conditions from early diagenesis (KI=1.0 and T about 100°C) to epizone (KI=0.21; T>300°C) in a very short interval of depths or for different grain size fractions in the same sample (e.g. dob-389). Authors should provide a geological explanation for such difference in temperature: It is clearly shown in Table 1 that KI=1.0 has been obtained for the finest size fraction (<0.1µm) of Dob-389.6, whereas KI of <2µm of the same sample is 0.43. However, KI=0.21 is obtained for the coarsest (2-10µm) size fractions of the deeper sample DOB-449.3. Nevertheless, we included a new discussion on the rapid mineralogical change in deeper part of the Dob well (see section 5.4 in the revised paper): “Particularly, samples deeper than 441 m consist of entirely 2M1 illite with KI values indicating epimetamorphic conditions, with corresponding illite precipitation temperatures of roughly 300°C or higher (Merriman and Frey, 1999). A prominent spike in KI values is apparent at 449.1 m (Fig. 10). This rapid mineralogic change indicates a significant increase in the paleogeothermal gradient in the lower part of Dobbyn 2. It is possible that abundant faulting and fracturing (this is also reported in the core logging report of Fitzell et al, 2012) induced significant hydrothermal circulation cells affected particularly the deeper section.”

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Re – Alternatively, do KI data record the maximum temperature conditions during faulting or hydrothermal activity, or they provide, in this case, misleading interpretation for very low grade metamorphic zonation? Are KI data reflecting a mixing of authigenic and detrital illite/muscovite populations? We already discussed in the original paper (5.1. Faulting, fluid-rock interactions and clay generation) that illite precipitation occurs during hydrothermal fluid circulation events triggered by faulting. We discuss in the revised paper (section 5.4) that KI of  $<2\text{ }\mu\text{m}$  fractions record maximum temperatures. In the discussion of K-Ar ages vs. KI values in section 5.4, we demonstrated that the plateau (consistent) ages of JC samples with different KI values and size fractions are geologically meaningful, because and this is possible only, if no detrital illites are mixed with authigenic illites. Although decreasing K–Ar ages with increasing KI values of Dob samples could be due to decreasing amount of detrital illite/muscovite with decreasing grain size, K–Ar ages for different size fractions and KI values of sample Dob-389.6 (2-1,  $<2$ , 1-0.5, and  $0.5\text{ }\mu\text{m}$ ), Dob-449.1 (2-0.5,  $<2$ , 1-0.5, and  $0.5\text{ }\mu\text{m}$ ), and Dob-476.6 (2-1 and  $<2\text{ }\mu\text{m}$ ) are consistent within analytical error. This, together with authigenic mineral textures of illites (Fig. 3), suggests that the presence of detrital muscovite in  $<2\text{ }\mu\text{m}$  fractions is unlikely. Please see the relevant discussion in the revised manuscript in section 5.4. Re – It would be also useful to introduce the boundary limits of very low grade metamorphic zones (Diagenesis-anchizone, anchizone-epizone) as calculated by Kübler and Árkai indexes in the CIS scale in the “XRD and SEM clay mineral analysis” section. This is done in the revised paper. Please see section 5.4. 4) It is not clear to me whether the X-ray diffraction patterns of oriented mounts in the supplementary material are air-dried or ethylene-glycol solvated tracings and the reason why just one tracing is shown. In my experience both air-dried or ethylene-glycol solvated patterns should be provided to see peak shape variations due to mixed layering, to the occurrence of other  $10\text{-}\text{Å}$  phases (paragonite, phengite, muscovite) that may broaden the 001 illite peak and affect KI measurements. I suggest to draw a figure with a selection of air-dried and ethylene glycol-solvated X-ray patterns for various grain size fractions and move it to the main text. Such a figure could show the deconvolu-

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tion process used to determine KI and AI measurements. More details on instrumental setting, step size, tube radiation ( $\text{CuK}\alpha$ ,  $\text{CoK}\alpha$ ) should be added in the supplementary material (figure caption or above the X-ray patterns). We conducted XRD analysis on oriented mounts for both air-dried or ethylene-glycol solvated samples. This is already mentioned in section 3.1. However, as we mentioned in section 4.3.1: “XRD analysis shows that 001 peak position of the illite does not change after ethylene glycol treatment, which indicates that smectite-like clays are not present or their content is insignificant (Srodon and Eberl, 1984). There is also no noticeable change in KI values after the ethylene glycol treatment of the samples. Therefore, we show air-dried tracings in the supplementary material, and this has been noted there. Details on instrumental setting, step size, tube radiation have already been given in section 3.1. Technical points

- 1) The term “crystallinity” should be avoided throughout the text because it is qualitative and depends on the type of order, the dimensional nature of periodicity and the technique involved in its measurement. For phyllosilicates and clay minerals, which are low-symmetry minerals with strongly anisotropic structures, various chemical compositions and random or ordered mixed layering, the meaning of “crystallinity” is ambiguous. Guggenheim et al. (2002) - clays and clay minerals, 50, 406-409, suggested that the use of a “crystallinity” index should be avoided and have to be referred to the name of the author who originally described the parameter (e.g., Kübler index for illite FWHM determinations, Árkai index for chlorite FWHM measurements). Please modify the text accordingly. We modified the text accordingly. Thank you for your useful comment and suggestion.
- 2) Analytical procedure for performing Kübler (KI) and Árkai (AI) index measurements should be given as many variables influence KI and AI determinations such as sample preparation, instrumental conditions, and the presence of other micaceous minerals in the samples.
  - a. Background information for slide preparation, cation saturation and density of oriented mounts should be provided
  - b. Have the Kübler and Árkai indexes been calculated from air-dried or glycolated samples?
  - c. How did you perform Kübler and Árkai index determinations? Has the full width at half maximum height been calculated by a deconvolution method, from raw

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tracing or after background subtraction? d. Please provide standard deviation for your data. e. Authors should provide the “raw data” as measured half-height peak widths as measured in their lab as well as the “calibrated” half-height peak widths as obtained using their own calibration curve. see Kisch et al. (2004) – schweizerische Mineralogische und Petrographische Mitteilungen 84, 323- 331 f. Please provide the linear regression equation used for calibrating Kübler index values against the CIS scale. Kisch et al. (2004) recommend reporting the calibration regressions used and the uncalibrated data in all papers reporting on the KI. Our paper has not been designed for a specific clay mineralogy-oriented journal and our topic does not deal primarily with methodology of clay mineral preparations, though it is important for obtaining a reliable data set. Therefore, we do not go much in detail for sample preparation techniques, since we followed principally Warr and Rice (1994) and Warr (2018), where the background information is given (except we did not use any chemical for saturation of clays). b. As we mentioned above, we used air-dried samples for KI calculations, since we tested that KI values of glycolated samples are not different. c. The full width at half maximum height has been calculated from raw tracing or after background subtraction, using the same equipment as the one reported in Warr (2018) (EVA software for Bruker D8 Advance diffractometer). d. Standard deviation for what? We measured each CIS standard 3 times and averaged, which gave very close KI values. e. We are not convinced of the need of providing the raw FWHM data (we kept only calibrated data). The calibration regression that we used is:  $\text{CIS-FWHM} = 1.3568 \times \text{CSIRO} - 0.0486$  ( $R^2 = 0.99$ ), from which the raw KI can be back calculated. Kisch (2004) questions the reliability of CIS-calibrated KI values and discusses a number of possible data-conversion errors as the cause of the broader CIS values. However, according to Warr (2014), “the differences largely to sample preparation effects not included in the calibration by polished rock slabs”. Warr (2014) further states: “The correlation made by Warr and Rice (1994) assumed Kisch’s half-peak-width measurements to be  $0.04^\circ$  narrower than Kübler’s (Neuchâtel) values, as stated in the literature (Kisch, 1990). As a result, this factor was added to the conversion equation accordingly. However, after both H.

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Kisch and B. Kübler later presented their half-peak-width values for the CIS rock-chip standards that required sample preparation, it became clear that Kisch's experimental values were, on the whole, broader than Kübler's, and not 0.04° narrower as published. This discrepancy introduced a mean error of 18% broadening to the CIS values, which accounts for most of the 23% difference in question (Warr, 2014).” 2) Mineral abbreviations should follow recommendations by the IUGS Subcommittee on the Systematics of Magmatic and Metamorphic Rocks. At the moment Acronyms used for minerals are not appropriate. For instance Kaolinite should be abbreviated in Kln, illite in Ill etc. For a complete list of mineral abbreviations see: Kretz, R. (1983): Symbols for rock-forming minerals. *Am. Mineral.*, 68, 277 - 279. Mandarino, J.A. (1999): *Fleischer's Glossary of Mineral Species 1999*. Eighth Edition, 1999. The Mineralogical Record Inc., Tucson, Arizona, USA. Mandarino, J.A. & Back, M.E. (2004): *Fleischer's Glossary of Mineral Species 2004*. Ninth Edition, 2004. The Mineralogical Record Inc., Tucson, Arizona, USA. We thank the reviewer for informing us about the standard mineral abbreviation recommendations. We made changes accordingly.

Line numbered comments (minor points) Line 34 or Line 39 – I suggest to add a recent paper concerning K-Ar dating and illite polytypism of clay gouges from two major orogen scale, long lived faults in north- ern Iberia useful for understanding the key thermotectonic stages of intraplate brittle deformation. Aldega, et al. (2019). Unraveling multiple thermo-tectonic events accom- modated by crustal-scale faults in northern Iberia, Spain: Insights from K-Ar dating of clay gouges. *Tectonics*, 38 (10), 3629-3651. Thank you, this is a good suggestion. We added this paper.

Line 61 – please provide reference (e.g., Balsamo et al. 2014 - The signature and mechanics of earthquake ruptures along shallow creeping faults in poorly lithified sediments. *Geology*, 42, 435-438. Sorry, we can't see any relevance of Balsamo et al. 2014 to this specific discussion in our paper.

Line 65 – replace “2M” with “2M1” Done, thanks.

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Line 66 – replace “40Ar-39Ar” with “K-Ar”. In addition I would suggest to replace the generic term “clay” with the more appropriate “illite-1M/1Md polytype”. 40Ar-39Ar is correct because all referred paper in this paragraph used 40Ar-39Ar, rather than K-Ar. However, we replaced “clay” with “illite-1M/1Md polytype”.

Lines 68 and 71– replace “2M” with “2M1” Thank you.

Line 81 – The Carpentaria basin is Cenozoic in age in Figure 1 whereas a Jurassic-Cretaceous age is reported in the text. Please correct. Not really. Cenozoic is yellow, whereas Eromanga-Carpentaria Basin is green. So, it is correct as it is.

Line 136 – I would replace “grade of diagenesis” with “anchizone” as Kübler Index values were originally used for determination of the anchizone. In diagenetic samples Kubler index measurements may be affected by mixed layering and KI values may provide misleading interpretations for very low grade metamorphic zonation (see Aldega et al., 2007 – Clays and Clay minerals 55, No. 5, 504–518.). Done, thanks.

Line 147- “polarizing microscope” is repeated twice. Thanks.

Line 212 – delete “were used as the calibration standard” Thanks.

Line 258 – delete “and” Thanks.

Line 263 – I would replace “clasts of phyllosilicates” with “phyllosilicate minerals”. It is unclear from the SEM picture if they are really clasts or small crystals of phyllosilicate minerals. Done. Thank you.

Line 279 to 284. I suggest to briefly describe differences or similarities between illite crystallinity data from different grain size fractions (e.g.  $<2\mu\text{m}$  and  $>2\mu\text{m}$ ) for detecting the metamorphic grade. Good point, thank you. Done.

Line 289 and 290- replace “2M” with “2M1” Done.

Lines 301-305- I would be more cautious to assign small rounded crystals detected by SEM images to illite-1Md polytype. The same illite polytype can occur in different grain

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size fractions and reflect different crystallization episodes. Are you sure that those small crystals cannot be higher grade illite crystals (2M1) reduced by cataclasis or a new generation of illite crystals due to multiple faulting episodes? Furthermore I would delete the assumption of the Ostwald ripening process in the result section. All deleted.

Line 310 – replace “high diagenetic” with late diagenetic or deep diagenetic (see Frey and Robinson textbook pag 70 – Low grade metamorphism, Blackwell science, 1999). Replaced as deep diagenetic.

Line 314 and 315 – dates of  $1099.6 \pm 3.0$  Ma and  $1106.8 \pm 2.5$  Ma reported in the text are missing either in table or in figure 6. We reported in the text wrong number, while those in Table 1 and Fig. 1 are correct. Now the text has been corrected. Thank you.

Please check Line 320 – K-Ar data are not shown in table 3 as reported in the text. This should be Table 1. Corrected, thanks.

Please check Line 336, 339, 345, 354, 359 – replace “fig. 7a” with “fig. 7b” All corrected, thank you.

Line 361, 363 - replace “fig. 7b” with “fig. 7a” Thanks

Line 362 and 363 – age clusters of 995 and 1115 Ma are missing in figure 7a. Please indicate them. Indicated. Thank you.

Line 375 - replace “table 4” with “table 3” Thanks.

Line 424 – I would add after “carbonate dominated sediments” . . .”and at different burial or deformation depths” Added. Thank you.

Line 424-431. Foliated cataclasites have been observed at very shallow depths in siliciclastic sediments (<500 m, e.g., Balsamo et al., 2014 - Geology, 42, 435-438) and carbonate rocks (< 2km; e.g., Smeraglia et al., 2016 - Journal of Structural Geology 93, 29-50) as the result of cataclasis, clay smearing and/or pressure solution-precipitation in presence of fluids during deformation. The authors might refer to these two pa-

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pers as well. This is a good suggestion. We added this sentence in our text with the suggested references. Thank you.

Line 442 – provide reference (e.g., Smeraglia et al., 2016 - Journal of Structural Geology 93, 29-50) Done.

Line 461-463- the reference provided is quite old. Authors might refer to more recent papers dealing with metasomatic alteration zones in extensional setting (e.g., Rossetti et al., 2011- Geological Magazine 148 (4), 558–579) and geothermal areas (e.g., Maffucci et al., 2016 – Journal of Volcanology and Geothermal Research 328, 84–95). Very useful references. Thank you. Added.

Line 472 and 491- replace “2M” with “2M1” Replaced.

Line 512- replace “high diagenetic” with “deep diagenetic” or “late diagenetic” sensu Merriman and Frey, 1999. Done.

Line 570, 576, 580, 588 – In these lines many toponym or names (e.g., smoke hill volcanics, Amadeus Basin, Stuart Dyke swarm, Arunta Block) are missing in figures. A more large scale structural map of north central Australia would help readers to follow the discussion section. This map could easily replace the inset in figure 1. We provided references for all these names and toponyms, and think that they can be referred to by readers. However, for the final stage of our submission, we can provide such a detailed map.

Table 1 – please provide the standard deviation for illite and chlorite crystallinity measurements. We measured KI and AI values several times only for some samples, not for all samples (how can we do it for so many samples and their different size fractions?). Therefore, we cannot provide standard derivation, sorry. I would replace “diagenetic” with “diagenesis”, and “2M” with “2M1” Done.

Figure 1 – authors should provide the age for the Mount Isa and Etheridge Provinces, Georgina Basins and Canoble depression as readers may be not aware of the age

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of Proterozoic-Ordovician basins in north-central Australia. We extended the regional geology section significantly, and provided all these age information (please see the revised manuscript).

Furthermore, I would add a line drawing of the seismic line 07GAIG1 to better follow the geological setting. Also, we added Fig. 1b showing the interpreted migrated seismic section for part of seismic line 07GA-IG1 across the Millungera Basin.

Figure 3 –kaolinite described in the text is not shown in figure 3 a and b. Please indicate it. In In the photo in Fig. 3 kaolinite is not clearly seen, but it is shown in Fig. 4. Figure 6 – please label with sample names each Ar-Age plot. Sample names are given now in the figure caption. Figure 7a – the curve of figure 7a shows the probability distribution of K-Ar and Ar-Ar ages. It is unclear to me why some age clusters have not been labelled. At least, age clusters at about 995 Ma and 1115 Ma should be labelled. Ages of 995 Ma and 1115 Ma have been labelled now.

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