

**Contrasting Mesozoic evolution across the boundary between on and off craton regions of the South African plateau inferred from apatite fission track and (U-Th-Sm)/He thermochronology**

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## **Additional Supporting Information (Files uploaded separately)**

QTQt model files

## **Introduction**

The enclosed supplementary information provides full details on the analytical methods used as well as graphical representations of AFT single grain ages and track length distributions. Also included are additional inverse thermal history models and the original QTQt data files (uploaded separately) that can be immediately used by other users to replicate the thermal history inversion.

## Text S1

### Apatite fission track ages – External Detector Method

During the spontaneous fission of  $^{238}\text{U}$  in apatite, damage to the crystal lattice occurs creating a spontaneous fission track. These spontaneous fission tracks accumulate in the apatite crystal over time and are readily quantified by counting tracks which intersect a polished surface under an optical microscope within a defined area. To estimate the abundance of parent isotopes (i.e.  $^{238}\text{U}$ ), fission of  $^{235}\text{U}$  is induced and a mica external detector is used to record induced fission tracks. As the neutron flux during irradiation is monitored using the standard glasses within the sample stack, the number of induced tracks in the mica can be counted and is representative of the  $^{235}\text{U}$  content of the grain.  $^{238}\text{U}$  can then be estimated as the ratio of  $^{235}\text{U}/^{238}\text{U}$  is constant in nature (1/137.88). Using the relative track densities in the AFT age can be measured using the following equation:

$$t = \frac{1}{\lambda_d} \ln\left(\lambda_d \frac{\rho_s}{\rho_i} \rho_d \zeta g + 1\right)$$

Where  $t$  = age;  $\rho_s$  = spontaneous track density;  $\rho_i$  = induced track density;  $\rho_d$  = track density in a dosimeter (a standard glass of known uranium concentration);  $g$  = geometry factor which defines the relationship between spontaneous tracks that intersect the polished surface and induced tracks; and  $\zeta$  = a constant of proportionality which includes the fission decay constant. The value of  $\zeta$  is calculated by each fission track analyst and provides a means of standardising the counting process. The external detector method (EDM) (Hurford and Green, 1982) permits individual grains to be dated, even in the presence of heterogeneous uranium distribution.

### Apatite (U-Th-Sm)/He Analysis

The majority of single grain AHe analyses were performed by M. Wildman at the Scottish Universities Environmental Research Centre (SUERC) with a smaller proportion of analysis at University College London (UCL). Similar ages and dispersion of ages of the Durango apatite standard were obtained from both facilities. AHe analysis for samples SR-17 and V-10 were analysed by V. Mackintosh at the University of Melbourne. Analytical details for these analyses can be found in Gleadow et al., 2015.

### Helium Extraction

A manual He extraction line was used at SUERC after Foeken et al. (2006). He extraction at UCL was performed using a Patterson Instruments automated He extraction system. Single crystal aliquots were packed in Pt foil capsules and placed in 2 mm deep holes in a Cu planchet. The Cu planchet sat within a stainless steel chamber, which was closed, securely tightened and moved into position under the laser. The chamber and flexible steel tube connecting it to the extraction line were then pumped clean using a combination of turbo molecular and ion pumps, which clean the line of atmospheric H and He and generates a vacuum of pressure  $<10^{-9}$  torr. Heating tape was placed around both the chamber and flexi-tube for at least 24 hours to remove background  $\text{CH}_4$  and  $\text{H}_2\text{O}$ . Leak tests were routinely carried out during and following the cleaning procedure of the extraction system to ensure there was no leak in the system. Cold blanks were systematically run to ensure that no contamination of the extraction system had taken place. Hot blanks involving the heating of empty Pt capsules were also performed to ensure the capsule itself is not a significant source of He. A diode

laser system ( $\lambda = 808 \text{ nm}$ ) was used to heat samples to c.  $800^\circ\text{C}$ . This method heats the crystal indirectly to a temperature that will allow diffusive loss of He but will not melt the crystal itself.

A HidenHAL3F quadrupole mass spectrometer and a Balzers quadrupole mass spectrometer were used for gas measurements at SUERC and UCL, respectively. He concentrations from samples are calculated against a calibration standard of  $^4\text{He}$  which is obtained by repeat measurements of the  $^4\text{He}$  standard throughout the analysis, typically with a reproducibility of (1 – 2%).

### U, Th and Sm analysis

Following helium extraction, aliquots were spiked with a calibrated solution with a known concentration of  $^{235}\text{U}$ ,  $^{230}\text{Th}$  and  $^{149}\text{Sm}$ , which allowed the relative abundances of  $^{238}\text{U}$ ,  $^{232}\text{Th}$  and  $^{147}\text{Sm}$  to be determined.  $\text{HNO}_3$  was added to the spike solution to completely dissolve the apatite. At SUERC, samples were analysed using an Agilent 7500ce ICP-MS while at UCL, a Agilent 7700x ICP-MS system was used. A blank vial of  $\text{HNO}_3$  and vials of a U standard with known  $\text{U}^{238}$  concentration were added to the ICP-MS run so that sample measurements can be calibrated.

### Apatite (U-Th-Sm)/He age calculation

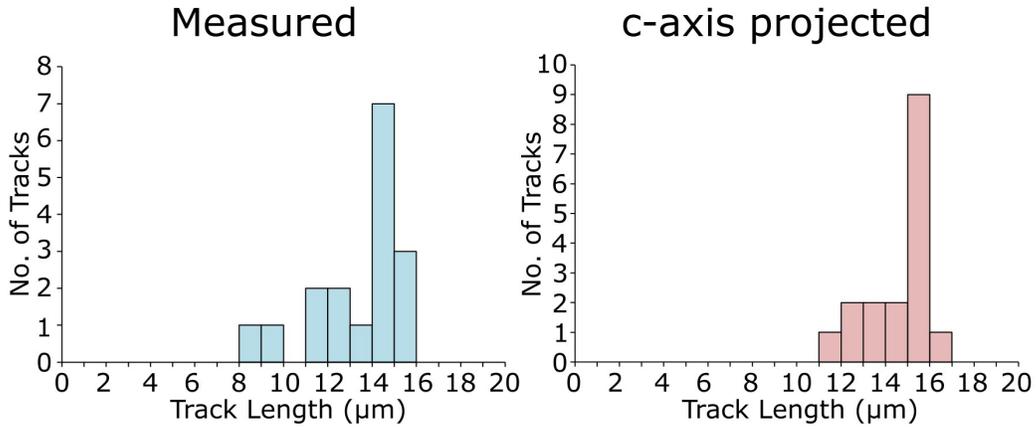
Once the amount of He, U, Th and Sm have been measured, the (U-Th-Sm)/He age can be calculated using the following equation:

$$[\text{He}] = 8 \left( \frac{137.88}{1+137.88} \right) [\text{U}] (e^{\lambda_{238}t} - 1) + 7 \left( \frac{1}{(1+137.88)} \right) [\text{U}] (e^{\lambda_{235}t} - 1) + 6 [\text{Th}] (e^{\lambda_{232}t} - 1) + 0.1499 [\text{Sm}] (e^{\lambda_{147}t} - 1)$$

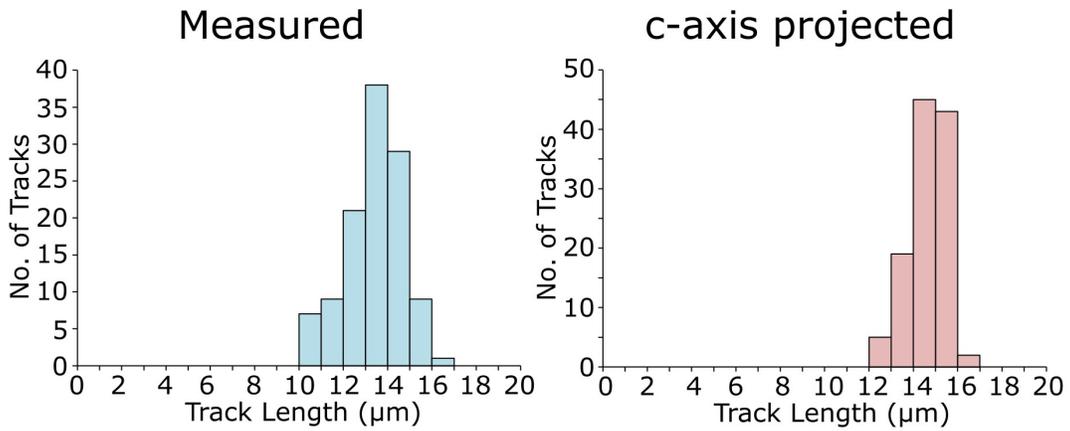
Where [U], [Th] and [Sm] are the concentrations of uranium, thorium and samarium respectively;  $\lambda$  is the relevant U and Th decay constant ( $\lambda_{238} = 1.55 \times 10^{-10} \text{ yr}^{-1}$ ;  $\lambda_{235} = 9.85 \times 10^{-10} \text{ yr}^{-1}$ ;  $\lambda_{232} = 4.95 \times 10^{-11} \text{ yr}^{-1}$ ,  $\lambda_{147} = 6.54 \times 10^{-12} \text{ yr}^{-1}$  and  $t$  is the age. The value of 137.88 is determined from the ratio of  $\text{U}^{238}$  to  $\text{U}^{235}$ , assumed to be constant in nature.

Figure S2

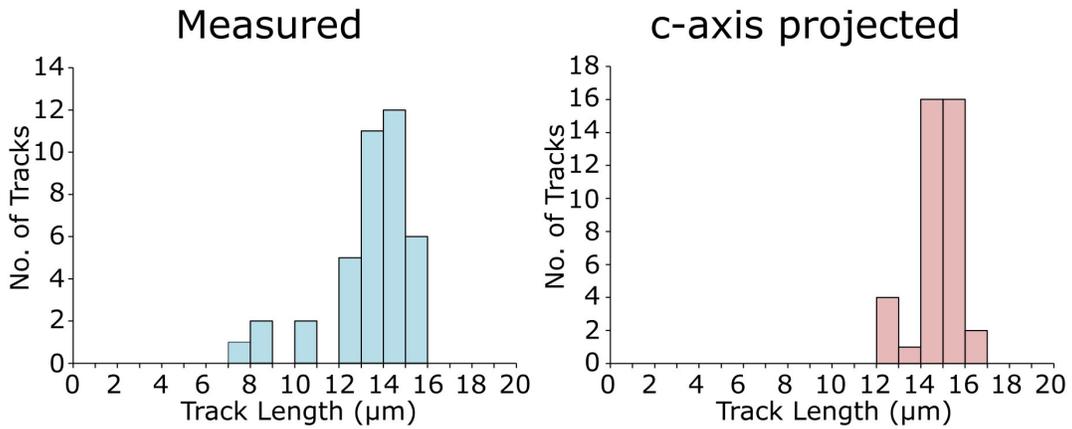
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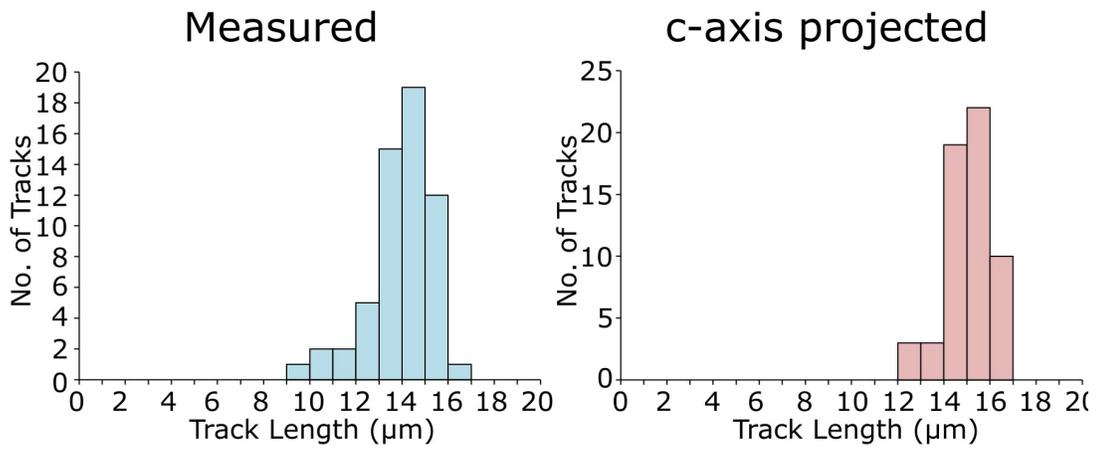
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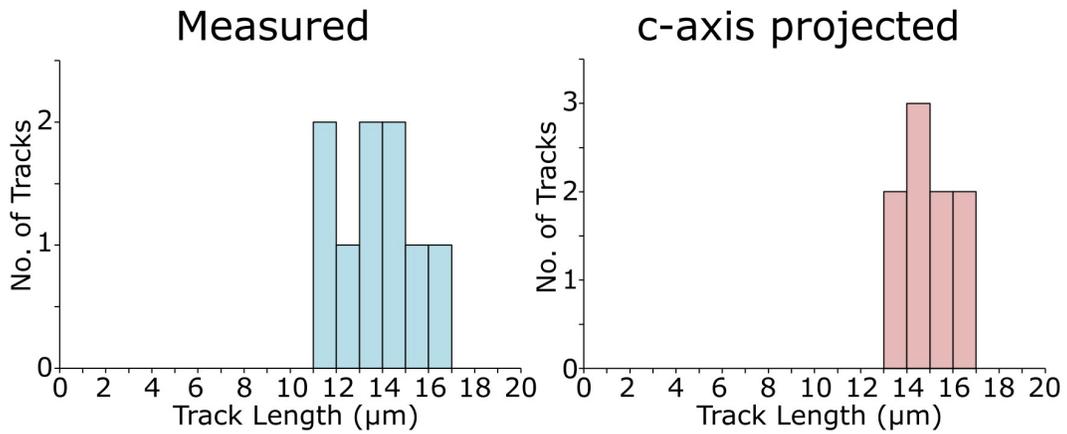
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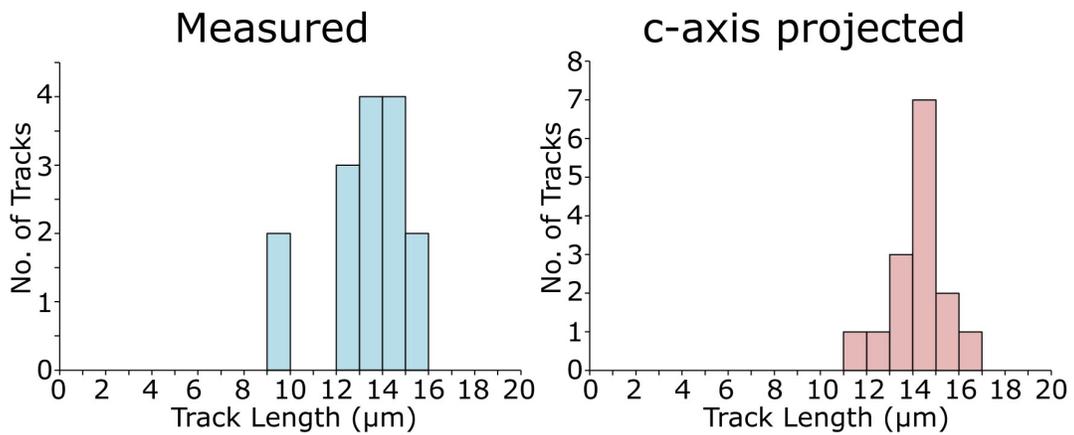
### SA12-09



### SA12-10

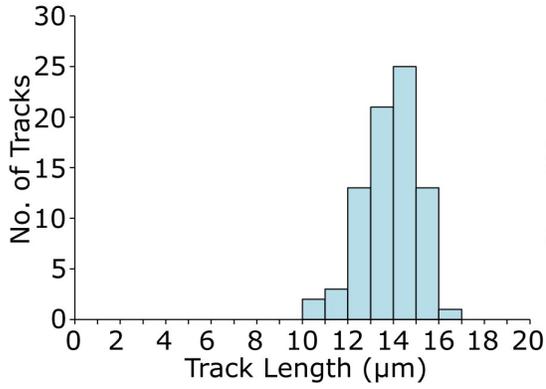


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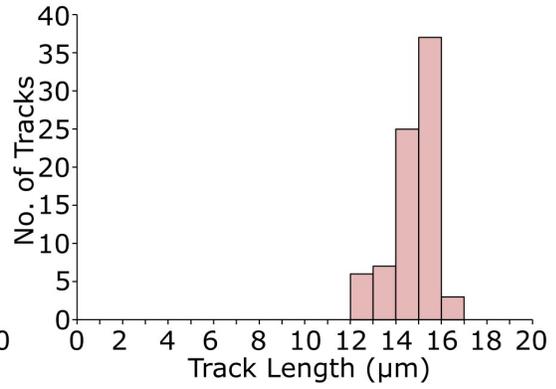


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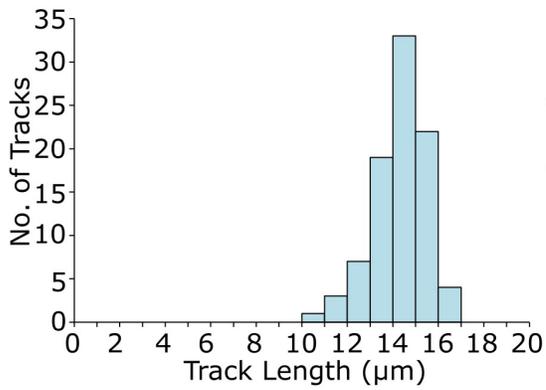


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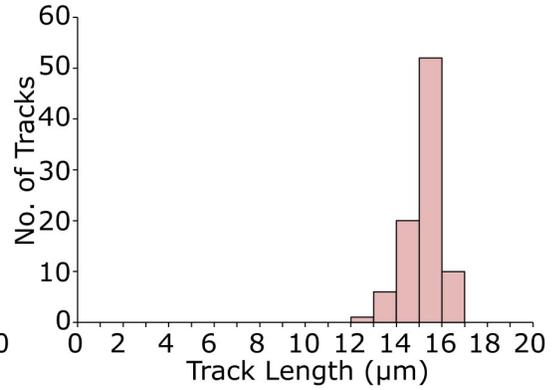


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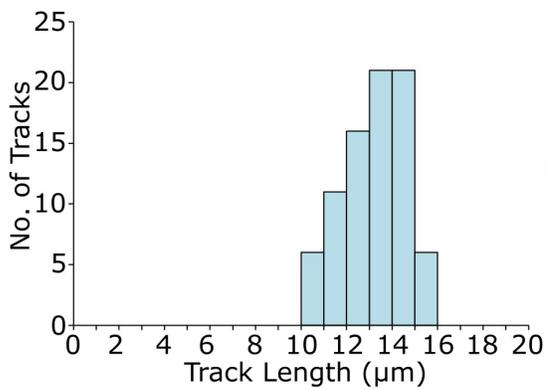


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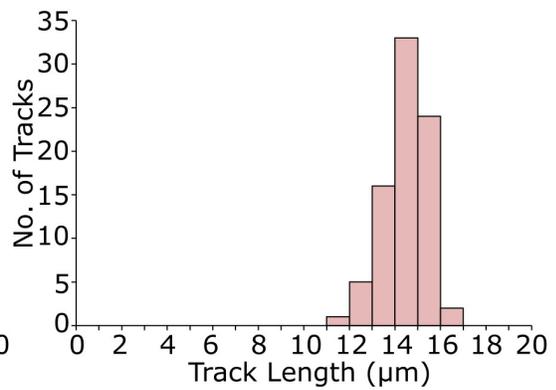


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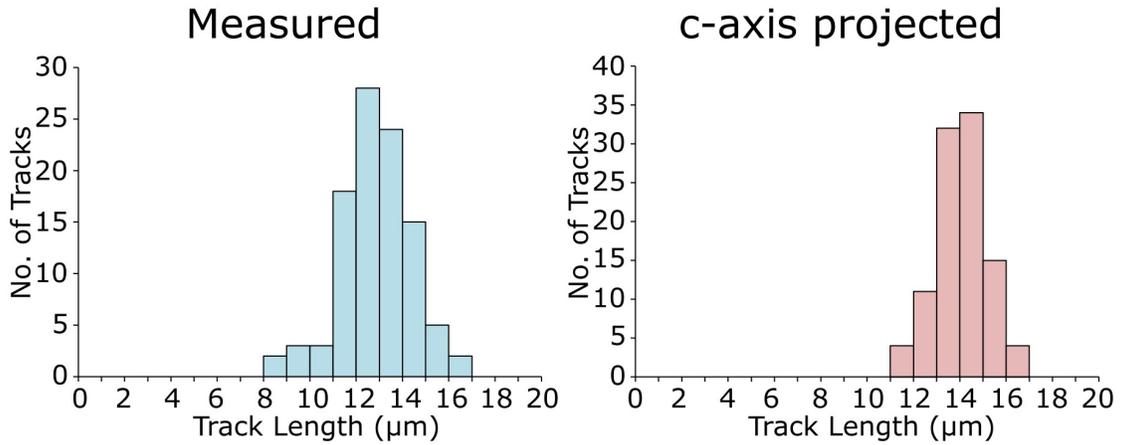
Measured



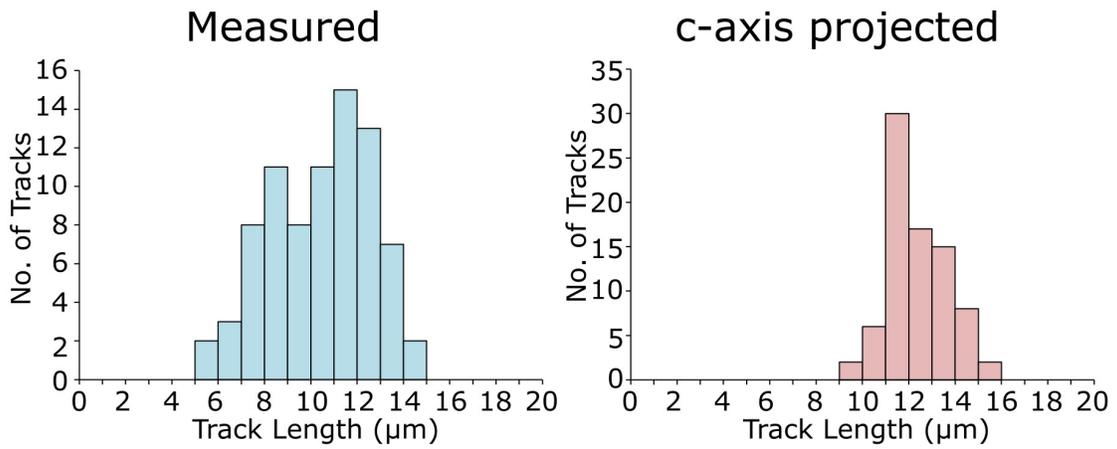
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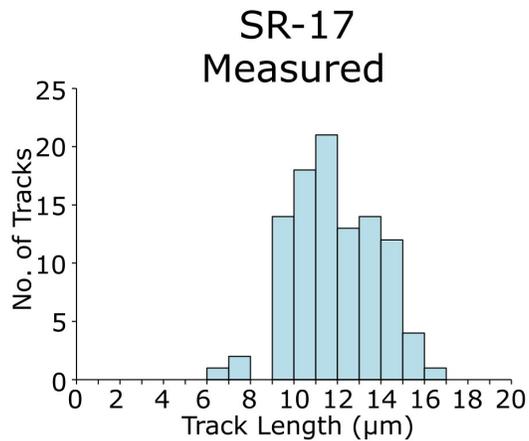
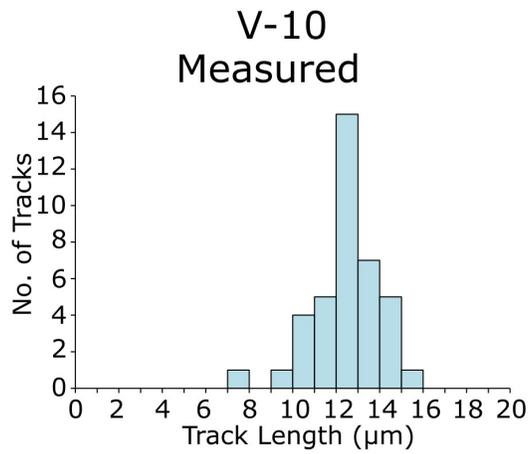
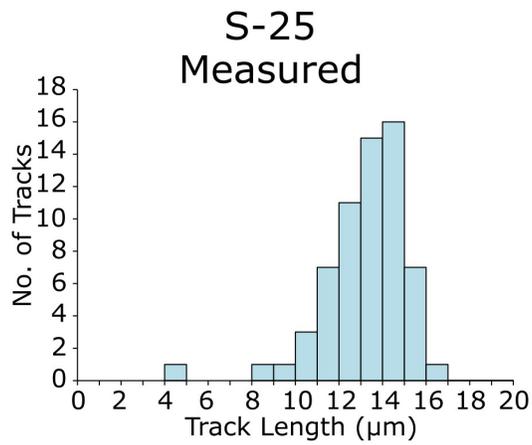


## GGO2



## PRU106

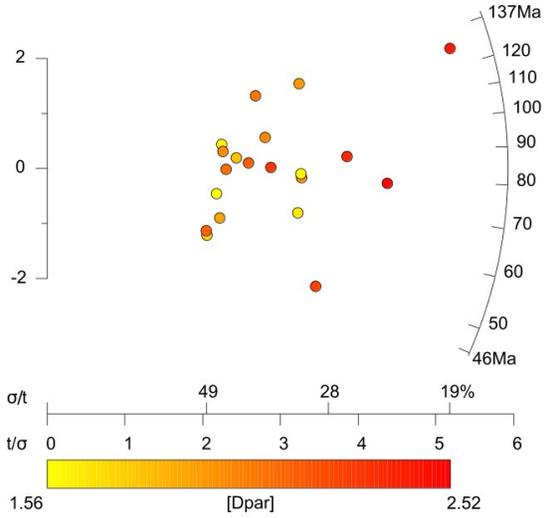




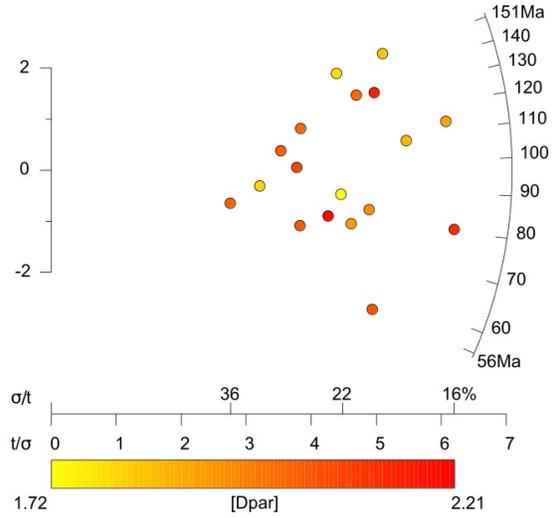
**Figure S2: Fission track length distributions for all analysed samples. Both raw track length distributions and track length distributions corrected for orientation to c-axis are shown except for S-25, V-10, SR-17, as these data were collected prior to routine measurement of the angle between measured track and c-axis.**

### Figure S3

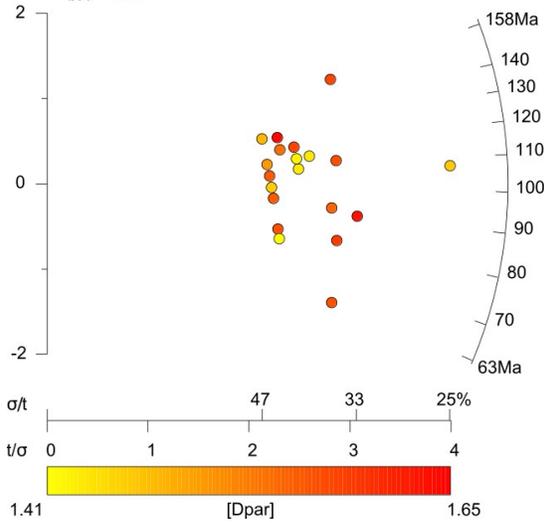
SA12-05 (n=20)  
 Central age =  $83.9 \pm 6.7$  Ma ( $1\sigma$ )  
 Dispersion = 13 %  
 $P(\chi^2) = 0.46$



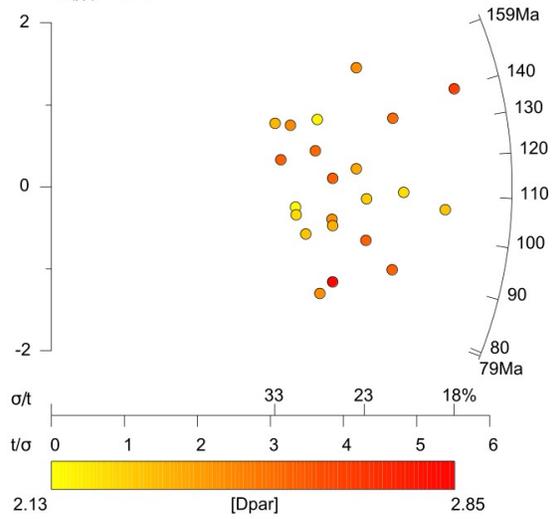
SA12-06 (n=18)  
 Central age =  $97.7 \pm 6.5$  Ma ( $1\sigma$ )  
 Dispersion = 18 %  
 $P(\chi^2) = 0.03$



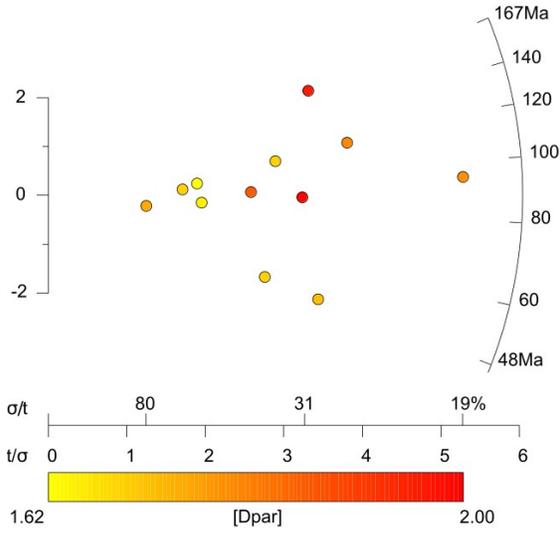
SA12-08 (n=20)  
 Central age =  $102.1 \pm 8.7$  Ma ( $1\sigma$ )  
 Dispersion = 0 %  
 $P(\chi^2) = 1.00$



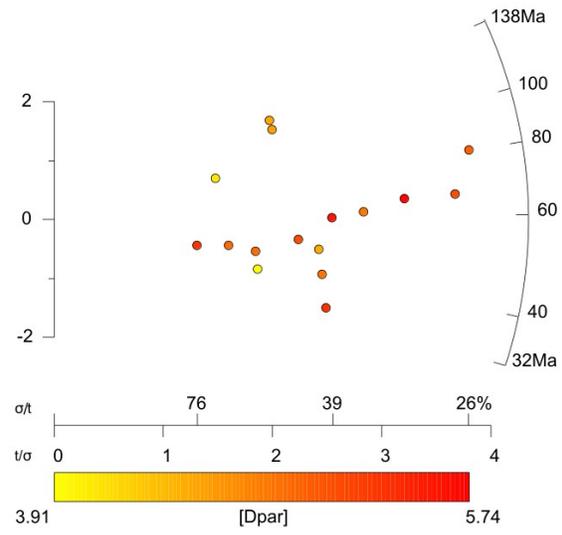
SA12-09 (n=22)  
 Central age =  $112.5 \pm 5.9$  Ma ( $1\sigma$ )  
 Dispersion = 0 %  
 $P(\chi^2) = 0.94$



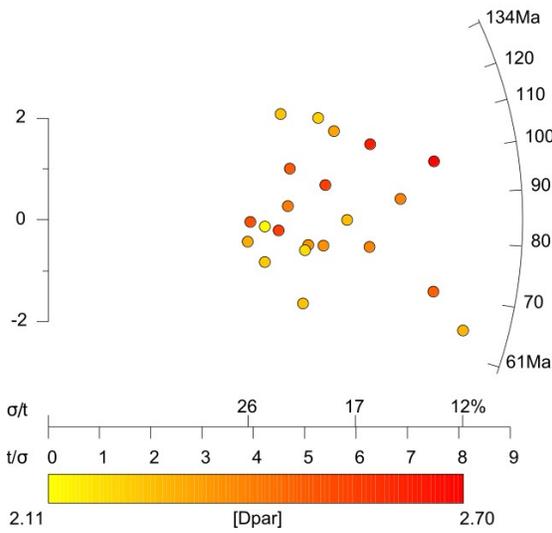
SA12-10 (n=12)  
 Central age =  $89 \pm 10$  Ma ( $1\sigma$ )  
 Dispersion = 21 %  
 $P(\chi^2) = 0.22$



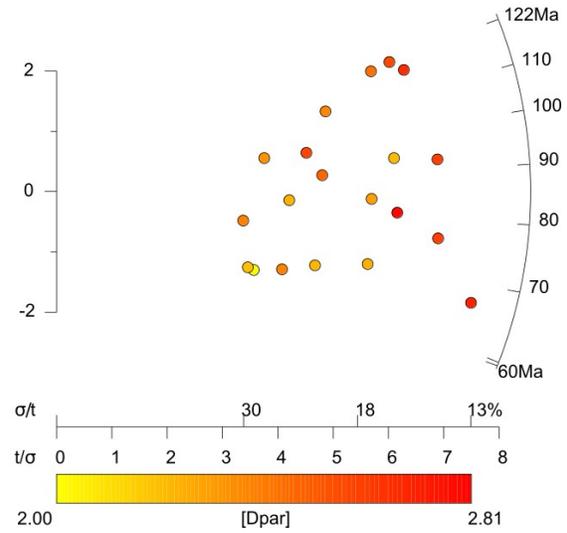
SA12-11 (n=16)  
 Central age =  $58.9 \pm 5.9$  Ma ( $1\sigma$ )  
 Dispersion = 0 %  
 $P(\chi^2) = 0.63$



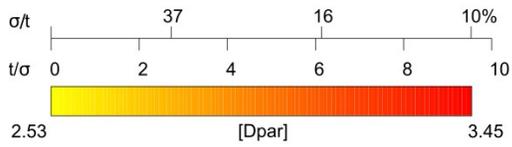
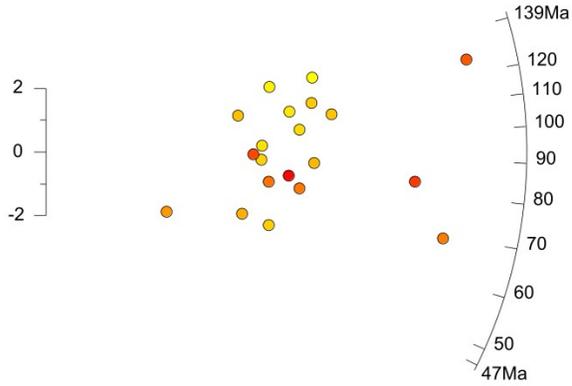
SA12-12 (n=22)  
 Central age =  $85.4 \pm 3.8$  Ma ( $1\sigma$ )  
 Dispersion = 10 %  
 $P(\chi^2) = 0.13$



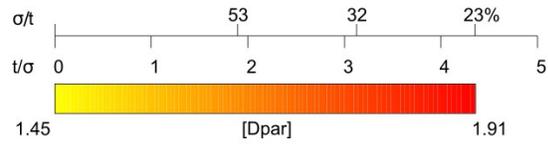
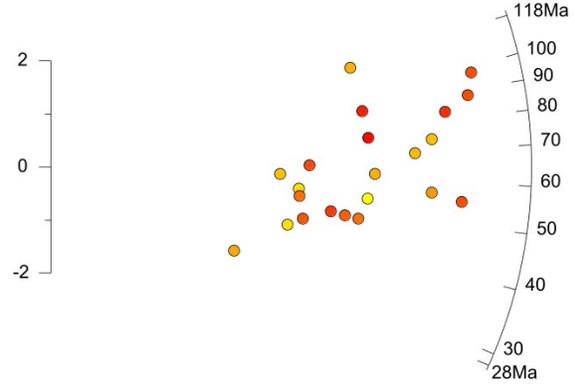
SA12-13A (n=20)  
 Central age =  $85.7 \pm 4.4$  Ma ( $1\sigma$ )  
 Dispersion = 12 %  
 $P(\chi^2) = 0.08$



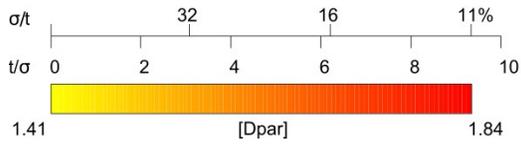
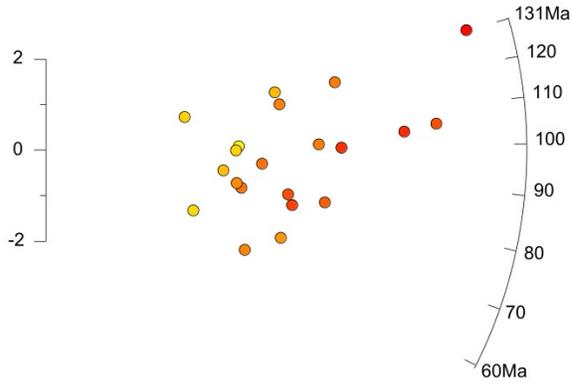
SA12-14 (n=20)  
 Central age =  $93.4 \pm 5.5$  Ma ( $1\sigma$ )  
 Dispersion = 20 %  
 $P(\chi^2) = 0.00$



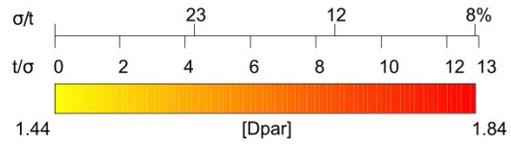
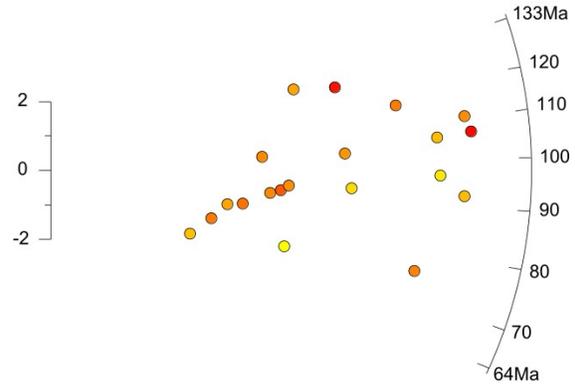
SA12-15 (n=22)  
 Central age =  $64.5 \pm 4.1$  Ma ( $1\sigma$ )  
 Dispersion = 0 %  
 $P(\chi^2) = 0.50$



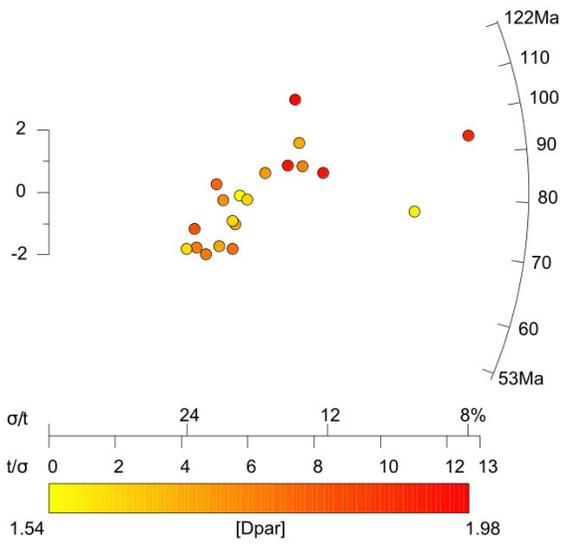
SA12-19B (n=21)  
 Central age =  $97 \pm 4.6$  Ma ( $1\sigma$ )  
 Dispersion = 12 %  
 $P(\chi^2) = 0.10$



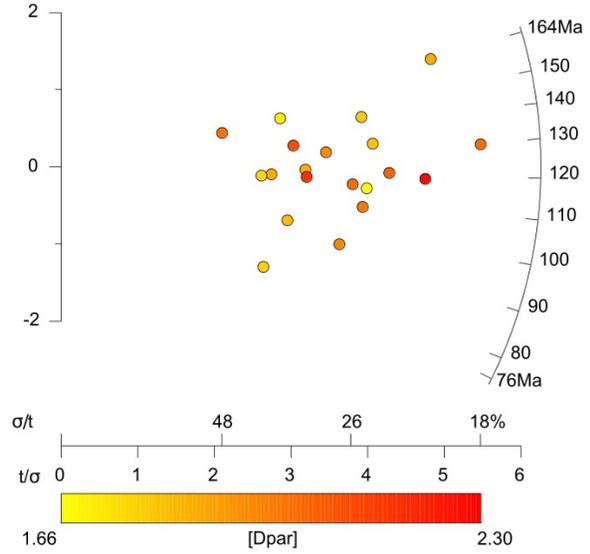
GGO2 (n=20)  
 Central age =  $96.6 \pm 3.6$  Ma ( $1\sigma$ )  
 Dispersion = 12 %  
 $P(\chi^2) = 0.00$



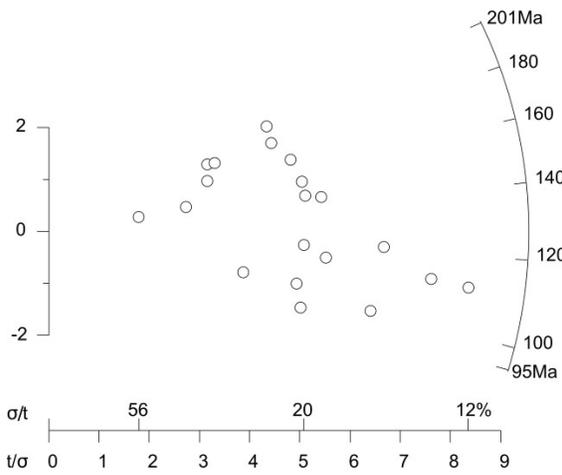
PRU106 (n=20)  
 Central age =  $79.6 \pm 3.8$  Ma ( $1\sigma$ )  
 Dispersion = 14 %  
 $P(\chi^2) = 0.01$



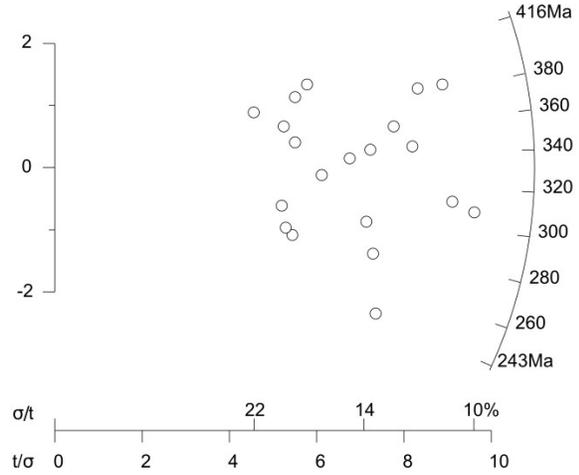
FS1605 (n=20)  
 Central age =  $122.8 \pm 7.5$  Ma ( $1\sigma$ )  
 Dispersion = 0 %  
 $P(\chi^2) = 0.99$



S-25 (n=20)  
 Central age =  $128.7 \pm 6.3$  Ma ( $1\sigma$ )  
 Dispersion = 8.4 %  
 $P(\chi^2) = 0.19$



SR-17 (n=20)  
 Central age =  $331 \pm 11$  Ma ( $1\sigma$ )  
 Dispersion = 2.4 %  
 $P(\chi^2) = 0.39$



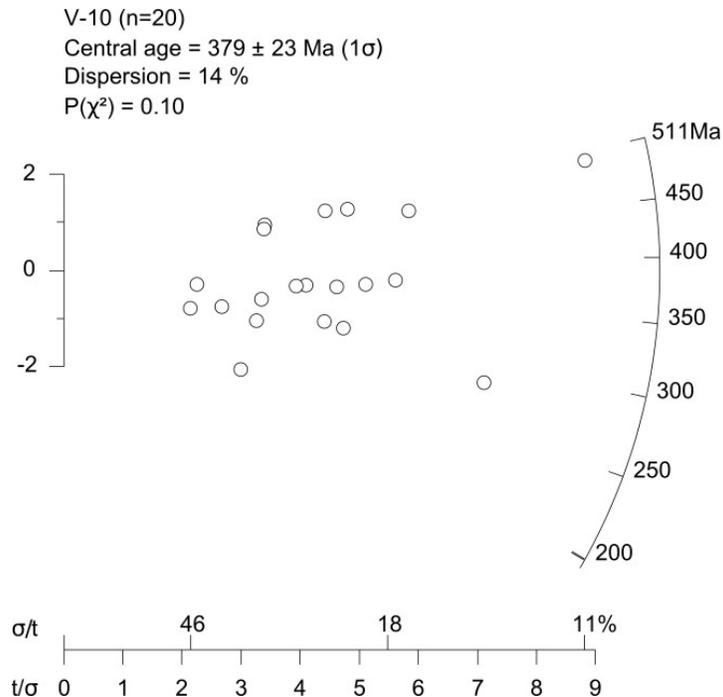
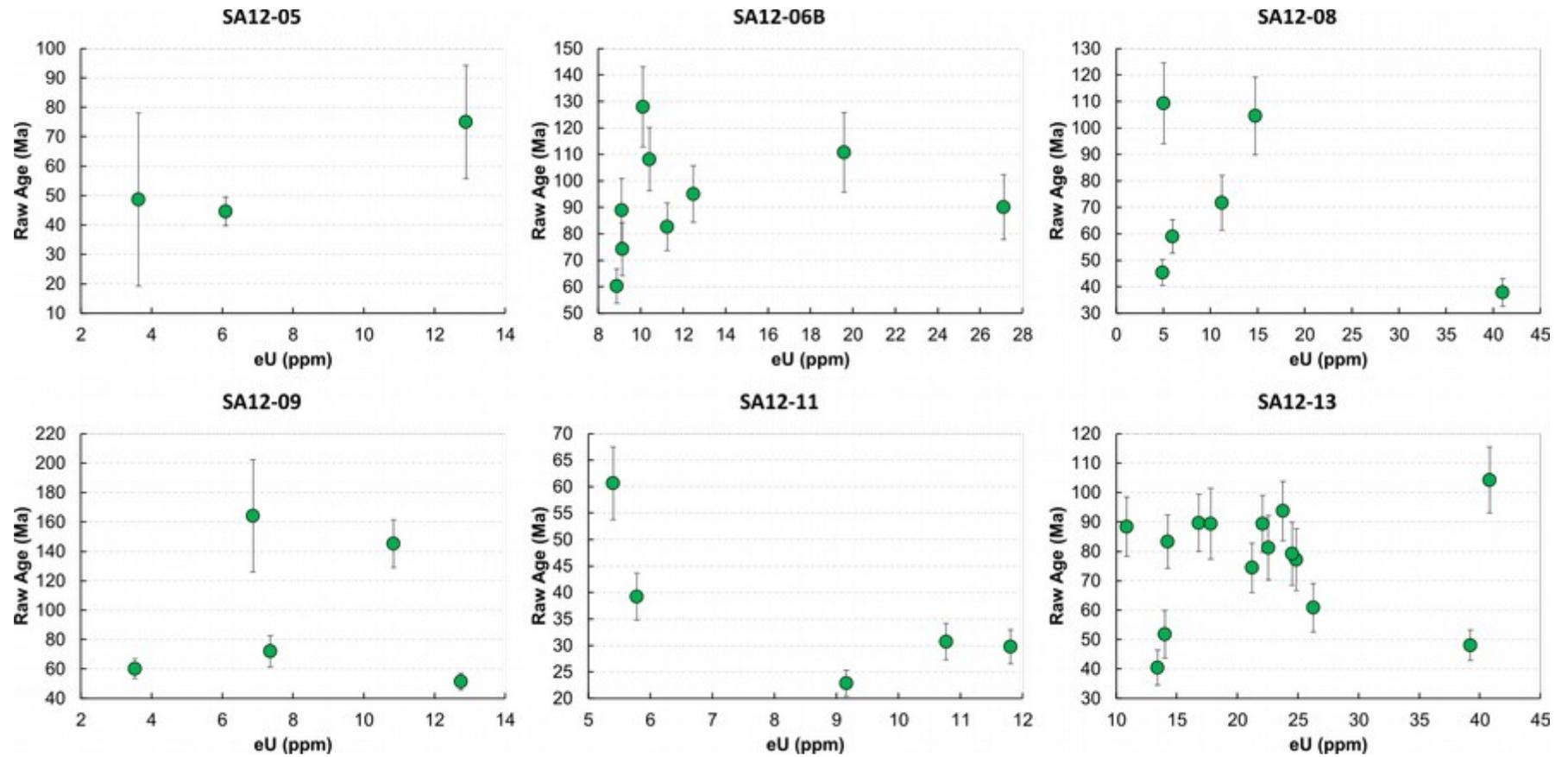


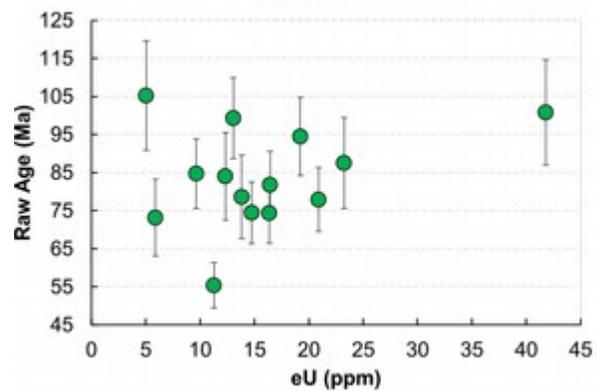
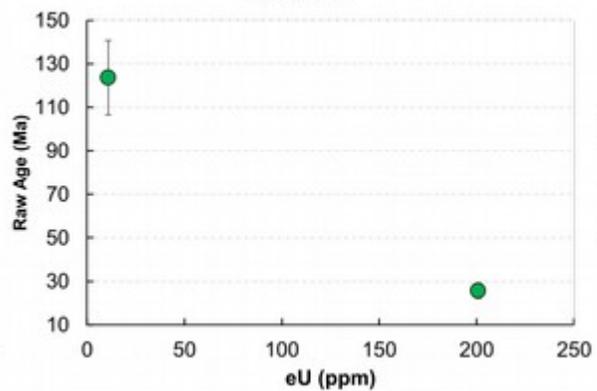
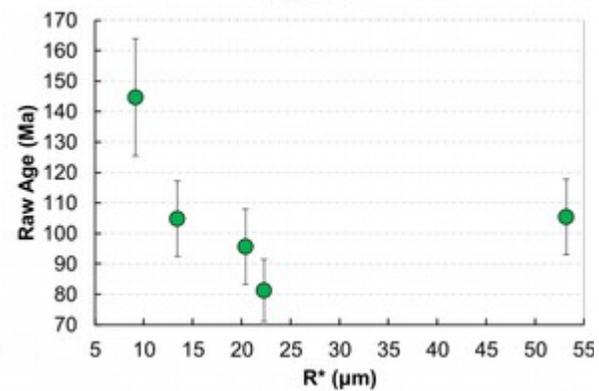
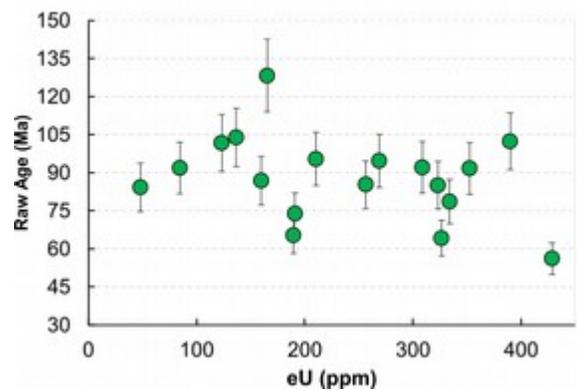
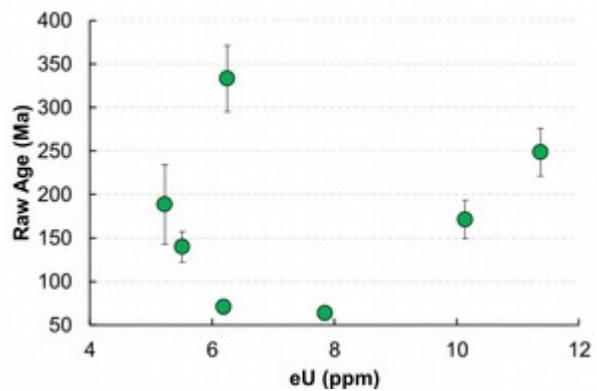
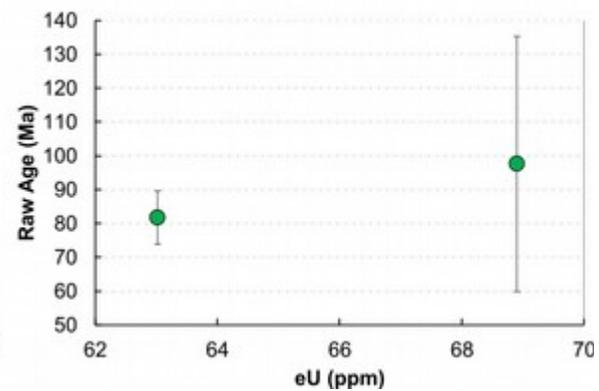
Figure S3: Radial plots of single grain apatite fission track analysis after (Galbraith, 2010). Radial plots were drawn using the Java application RadialPlotter (Vermeesch, 2009).

Plotting single grain AFT ages as a radial plot provides a means to display single grain age dispersion while taking into account the precision of each measurement (Galbraith, 2010). An AFT age ( $z$ ) with standard error  $\sigma$  is plotted as the point ( $x, y$ ) where  $x = 1/\sigma$  and  $y = (z - z_0)/\sigma$  for some  $z_0$  chosen so the average  $y$  is near 0.  $z_0$  is the transformation of central AFT age (a logarithmic transformation). Less precise data will plot closer to the origin than more precise data. Each single grain age can be determined using a line which cuts through the origin and the corresponding data point.

A  $\chi^2$  test for statistical homogeneity was performed on every sample with the corresponding p-value shown each radial plot. When the p-value is  $< 0.05$ , there is the possibility that the sample set contains more than one population. In these cases a mixture model (Galbraith and Green, 1990) was used to investigate whether the single grain age distributions better represent more than one population of ages. Radial lines on the radial plot indicate peak centres and the age of the peaks and proportions of grains belonging to that population are beneath the radial plot. Details of the algorithm used for the mixture modelling can be found in Vermeesch, (2009).

Figure S4 (a)



**SA12-14****SA12-15****SA12-19****GGO2****FS1605****PRU106**

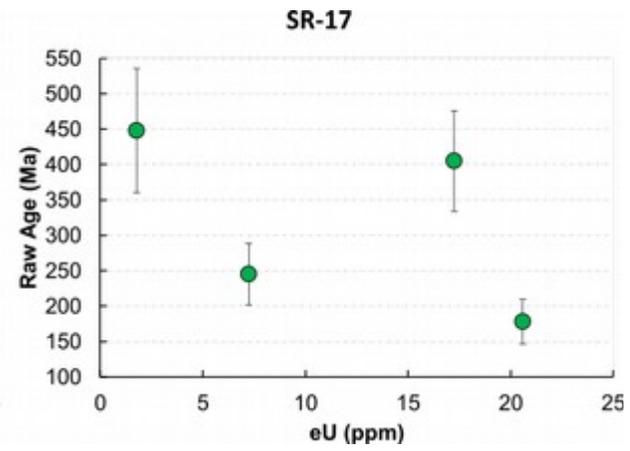
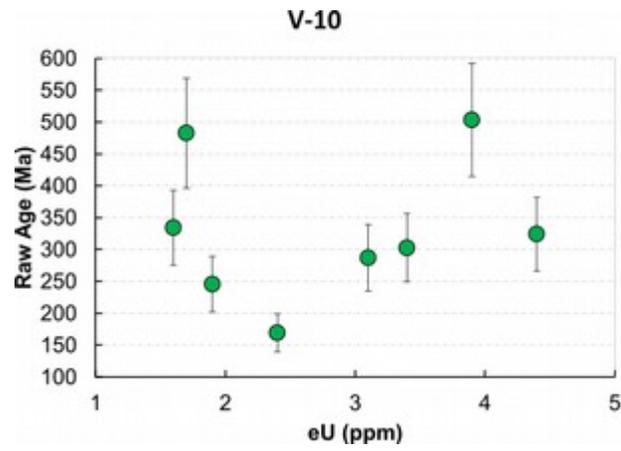
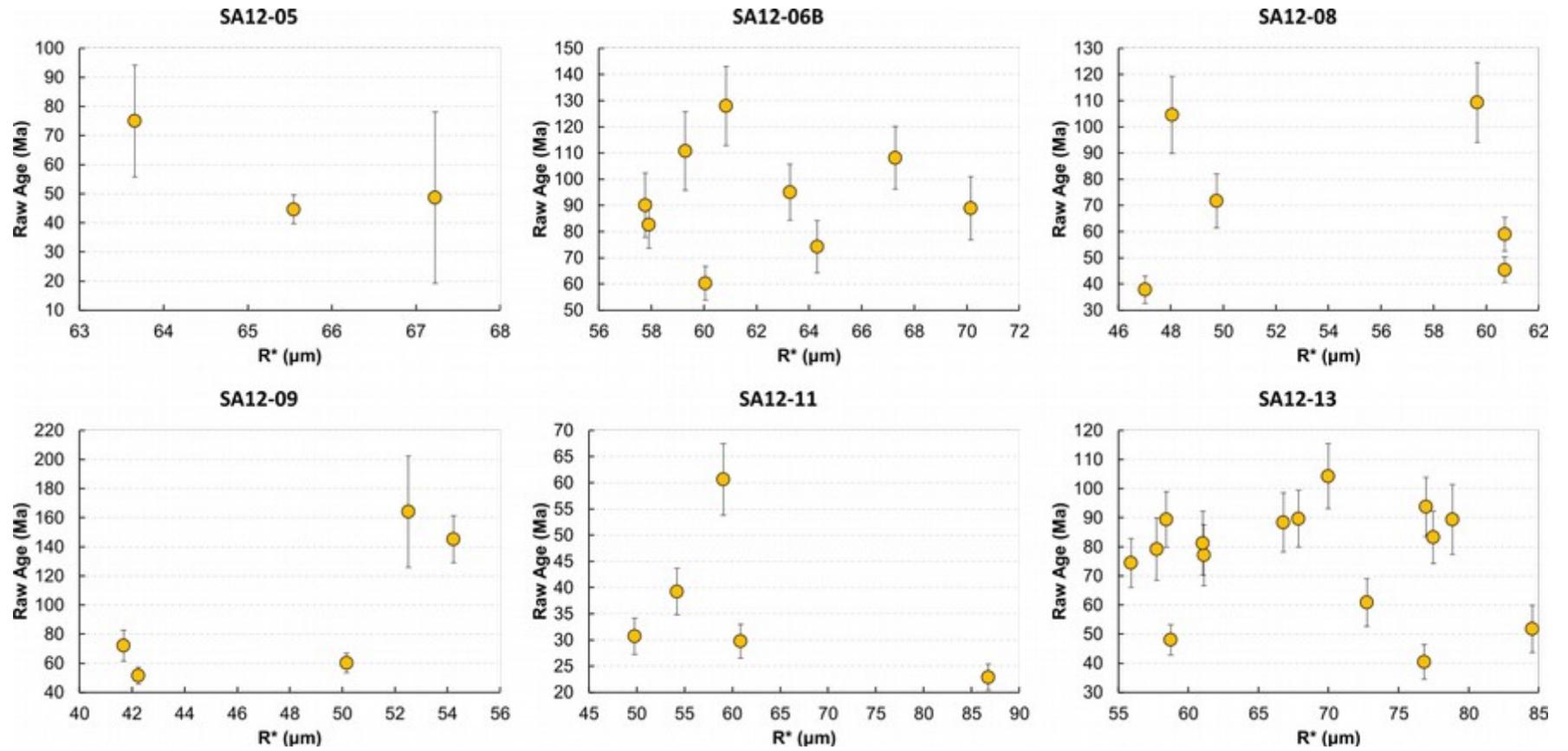
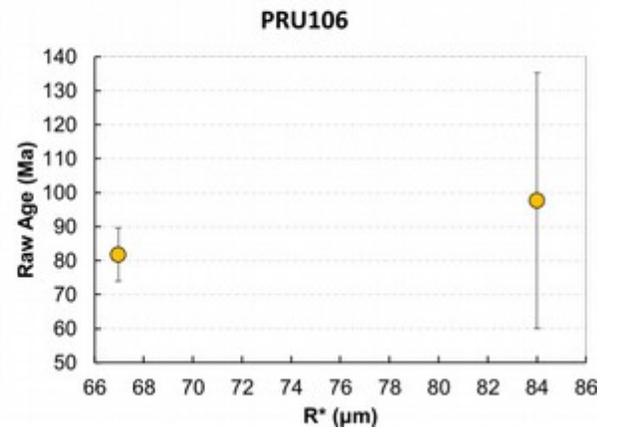
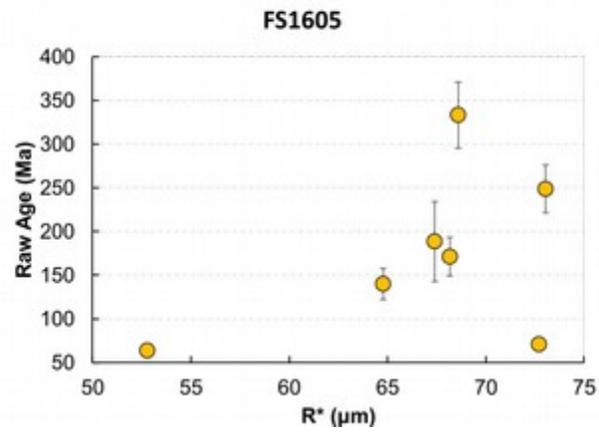
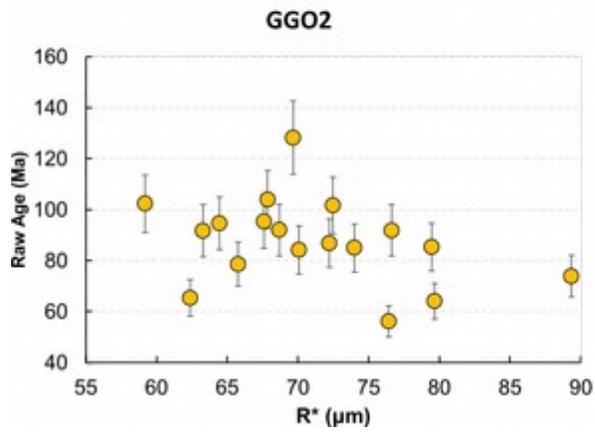
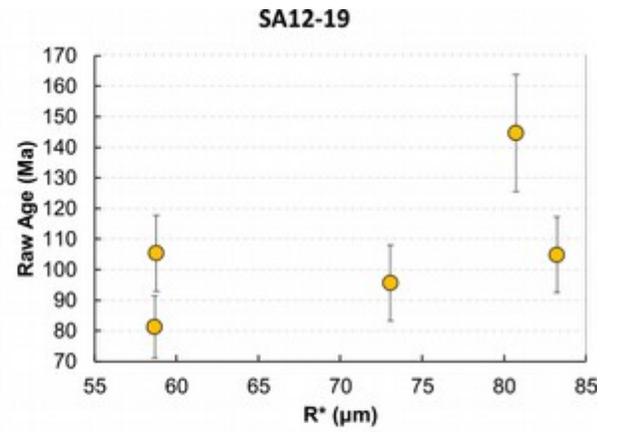
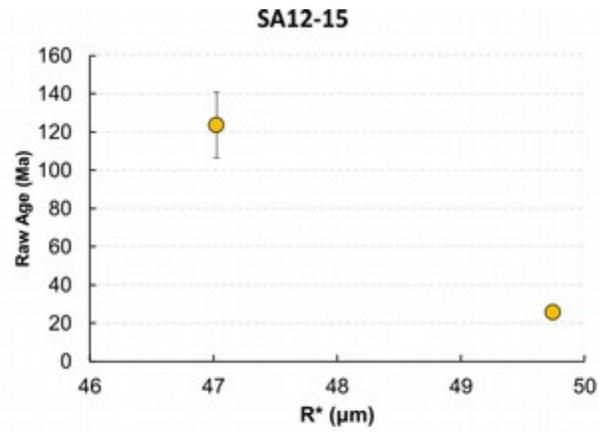
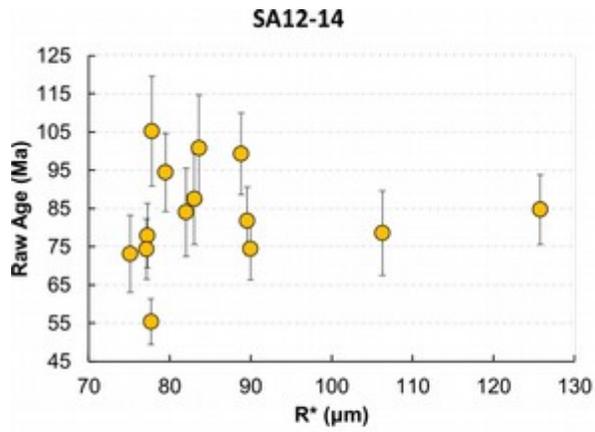


Figure S4 (b)





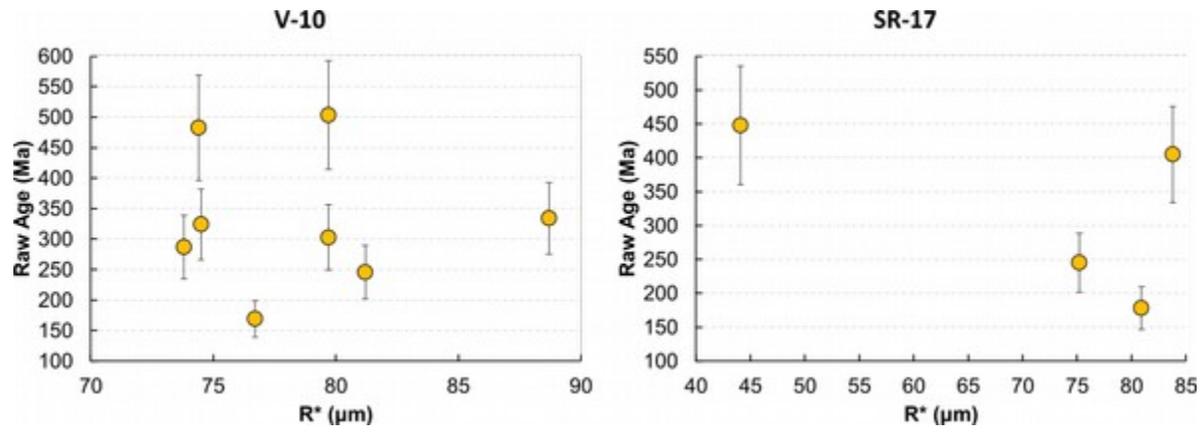
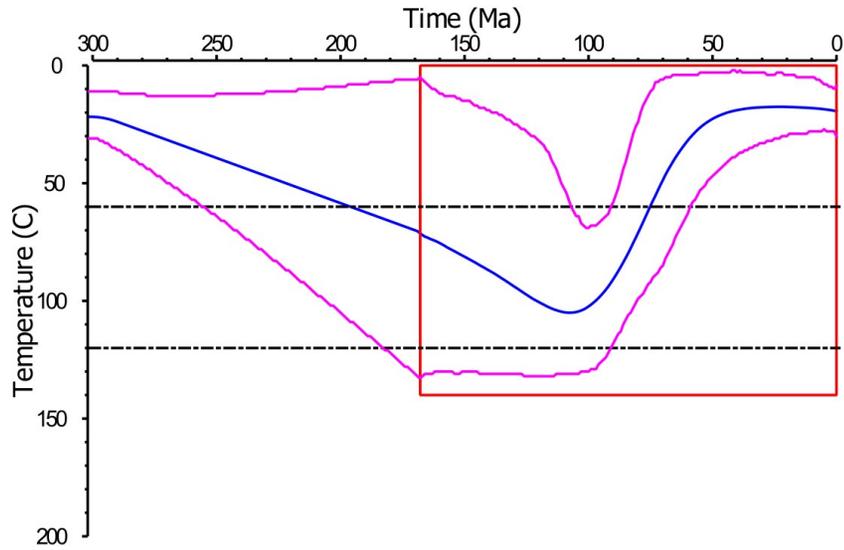
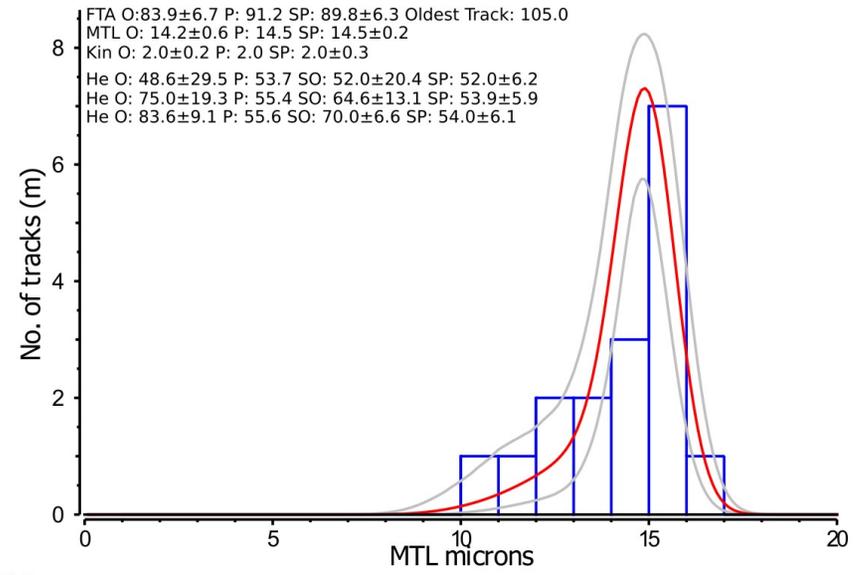


Figure S4: (a) AHe age versus effective Uranium content ( $[eU] = [U] + 0.235*[Th]$ ); (b) AHe age versus equivalent spherical grain radius.

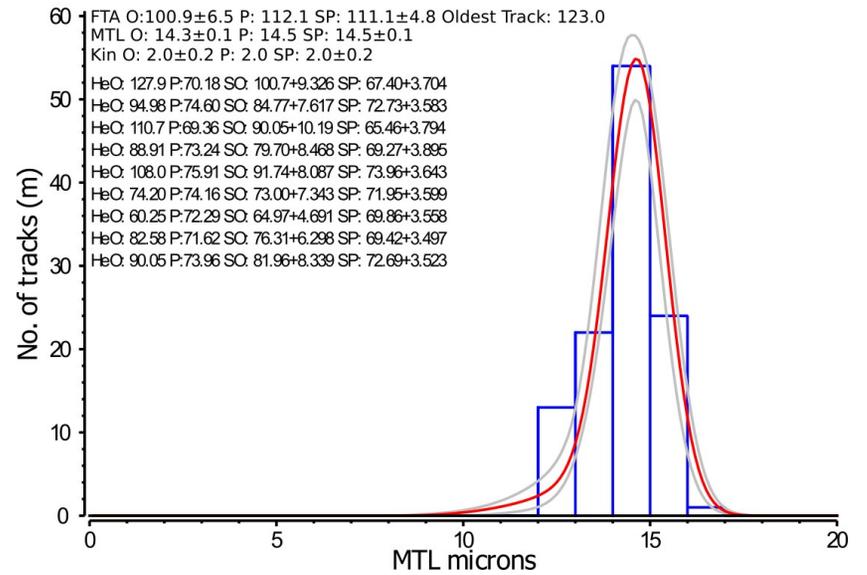
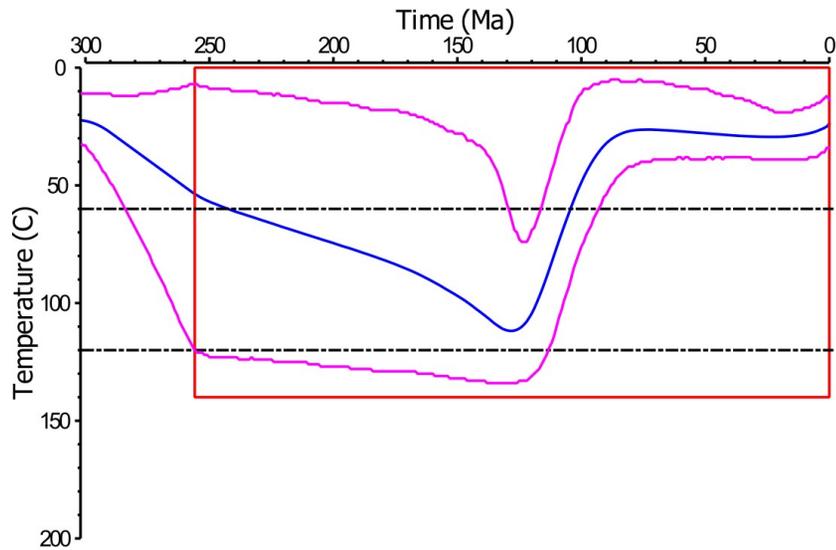
Figure – S5

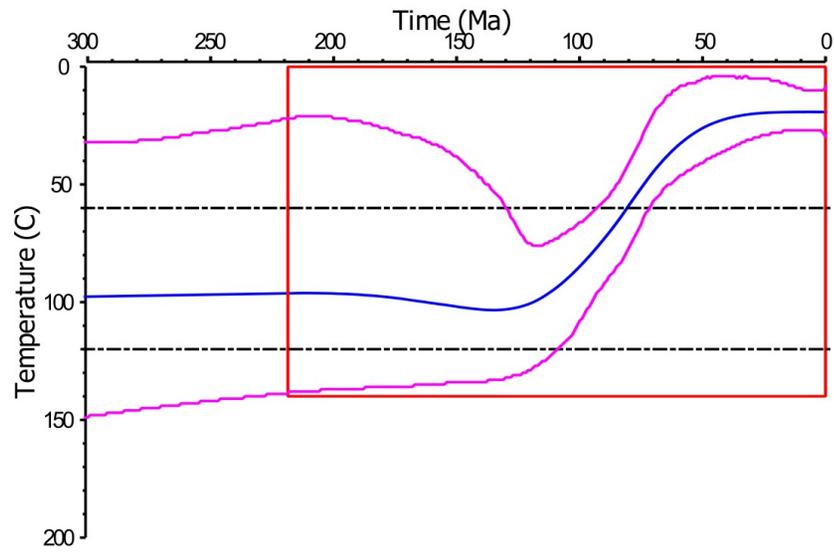


SA12-05

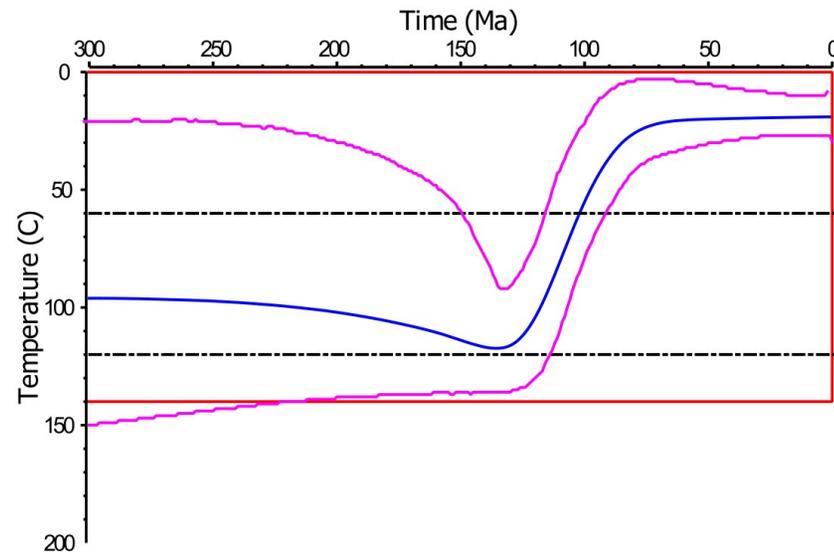
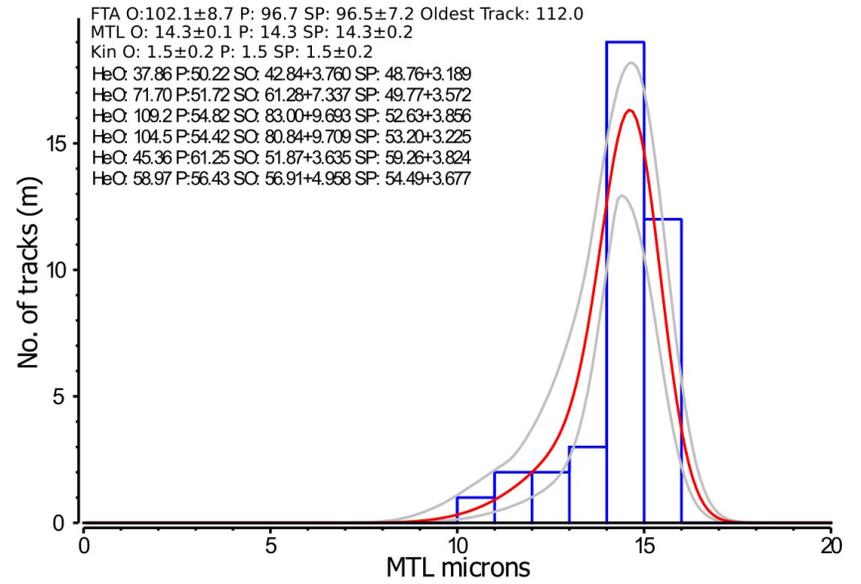


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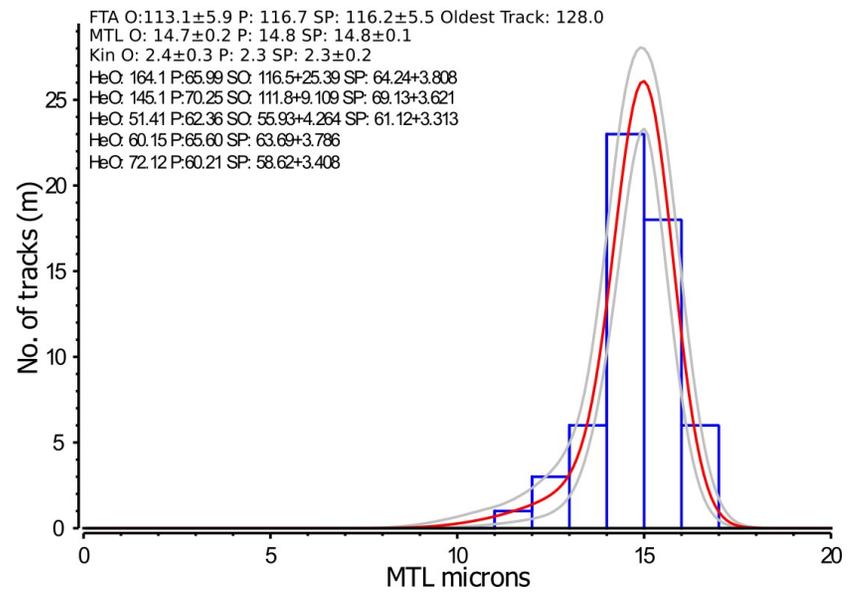


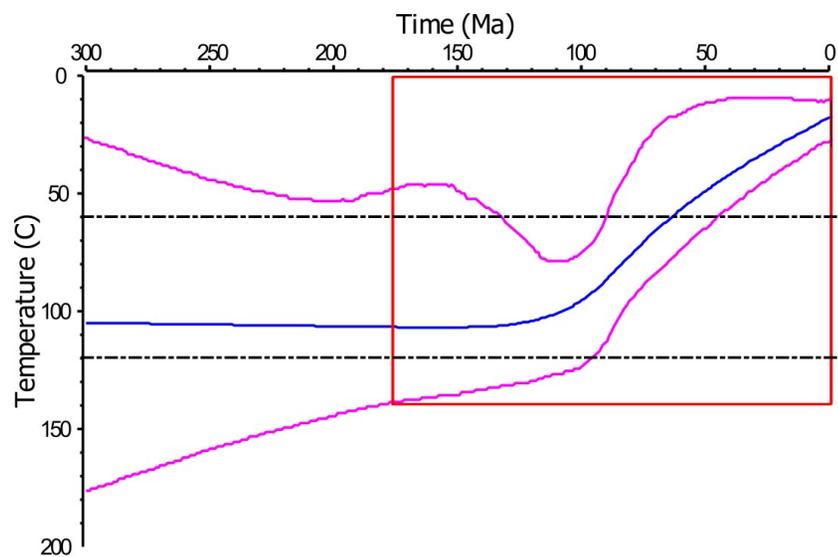


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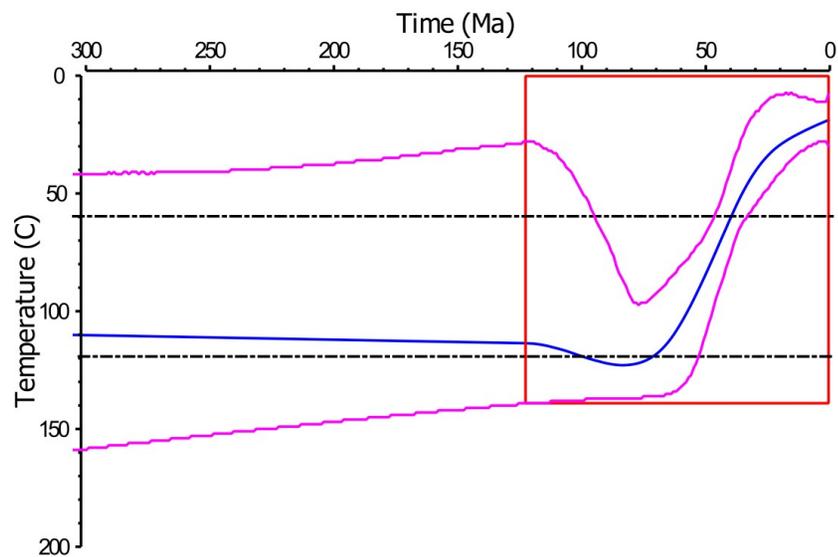
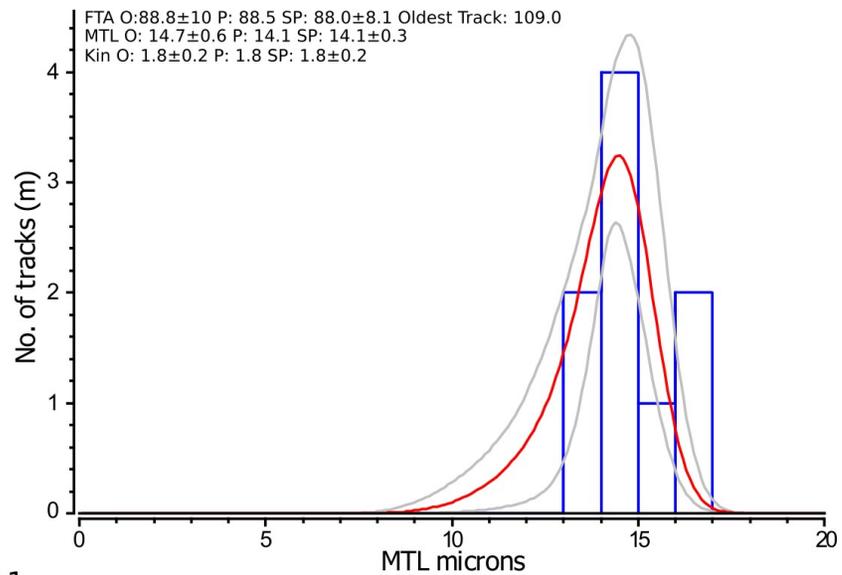


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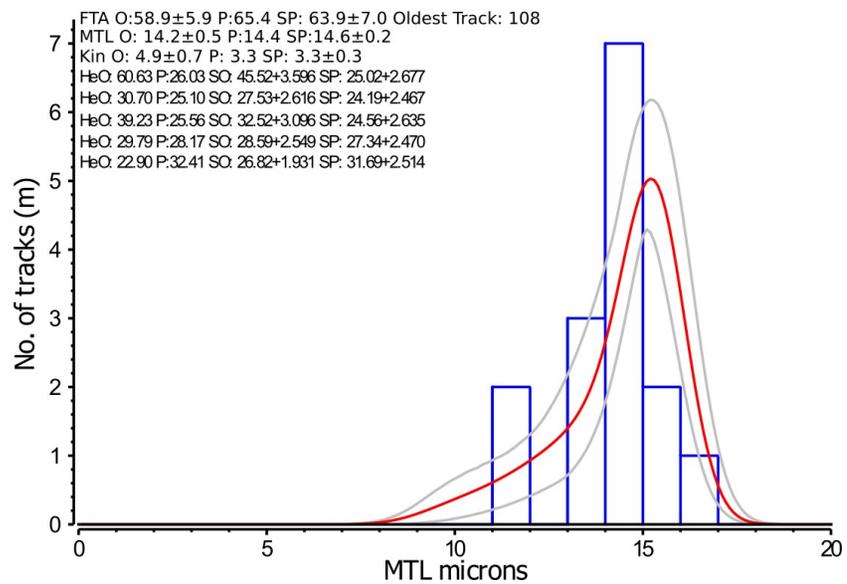




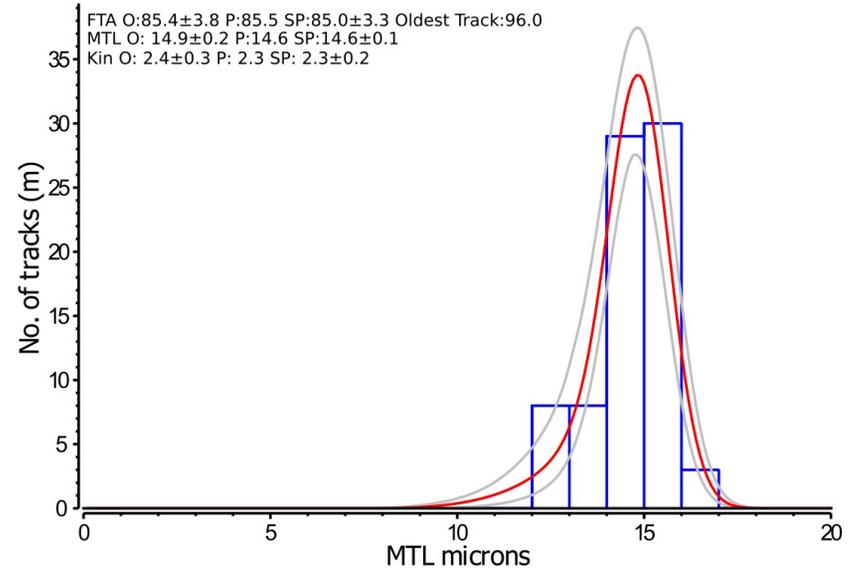
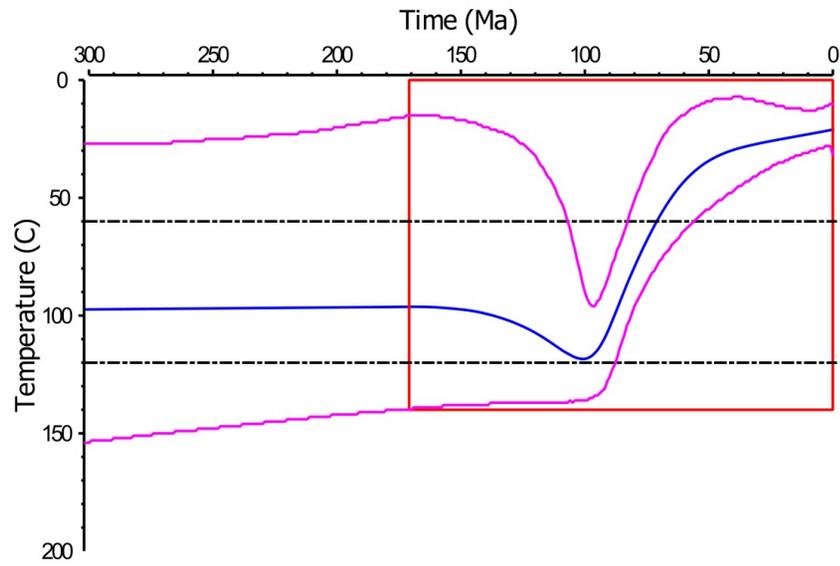
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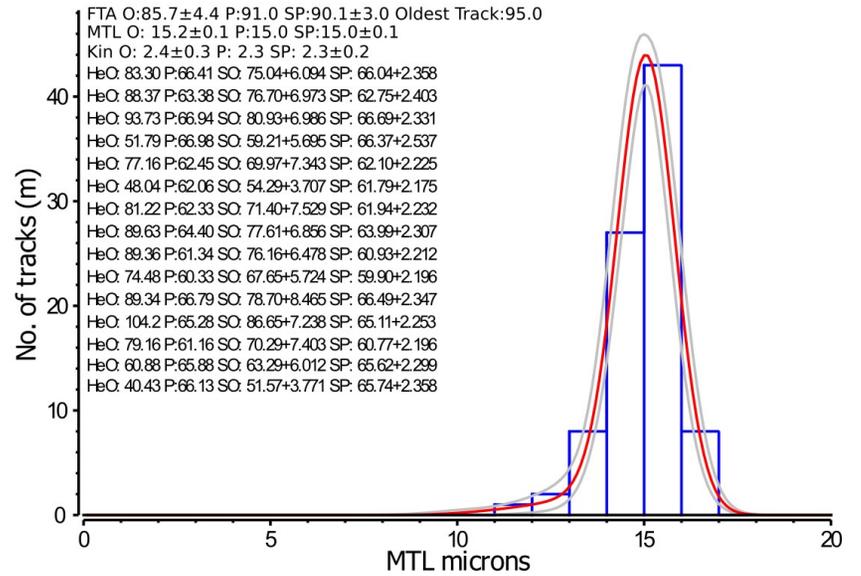
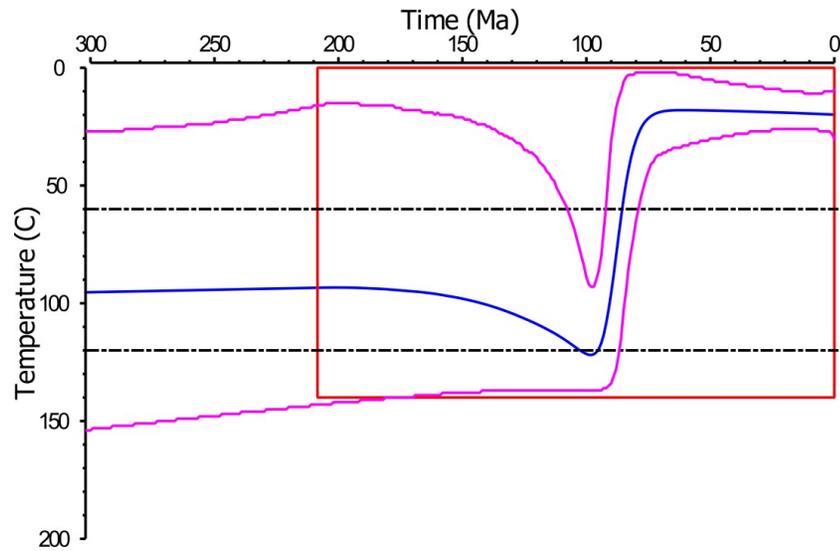
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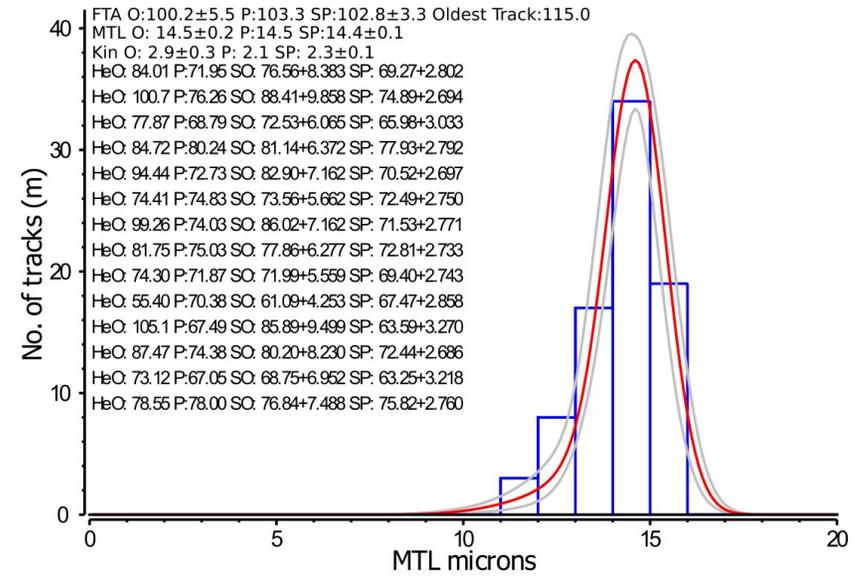
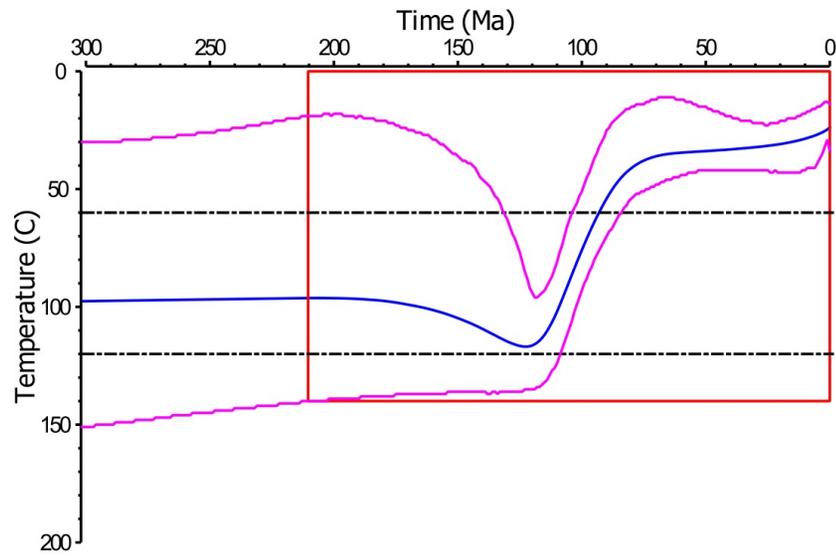
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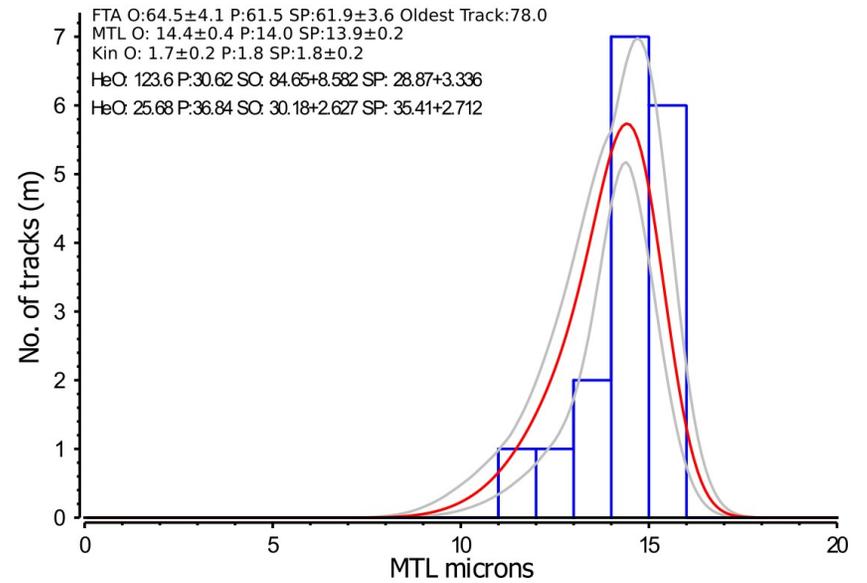
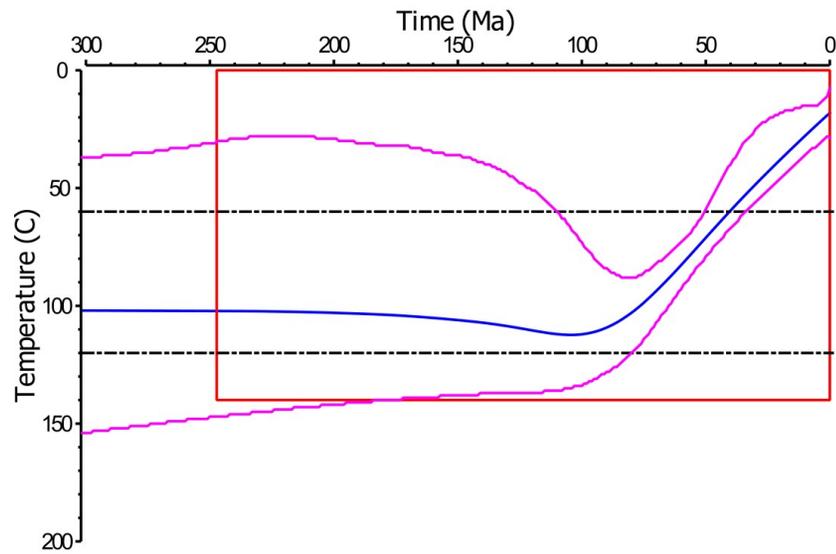
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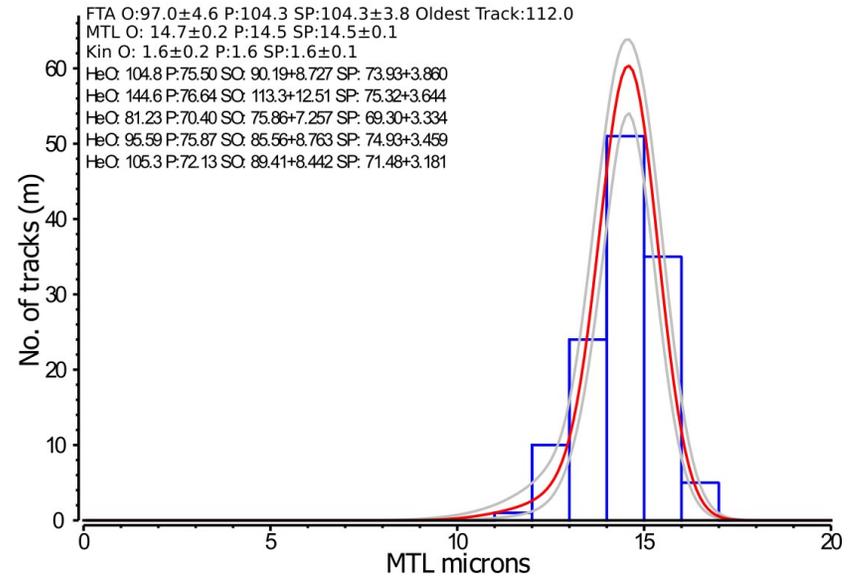
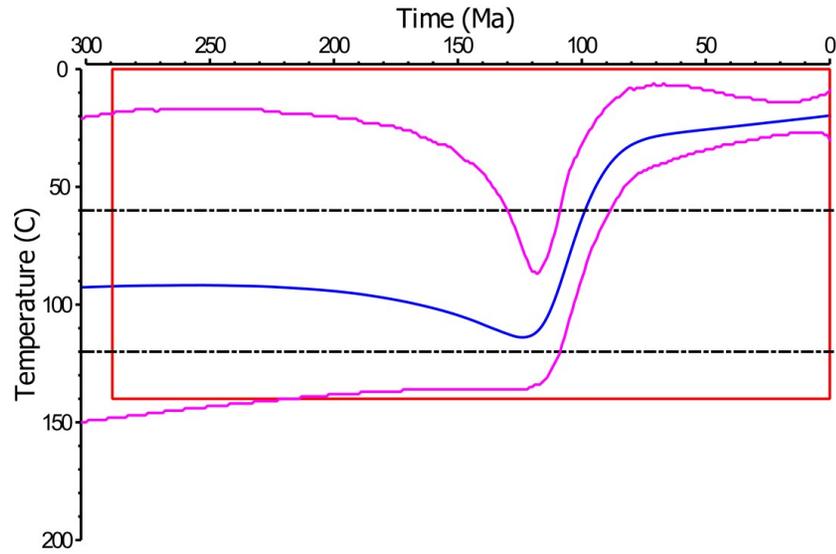
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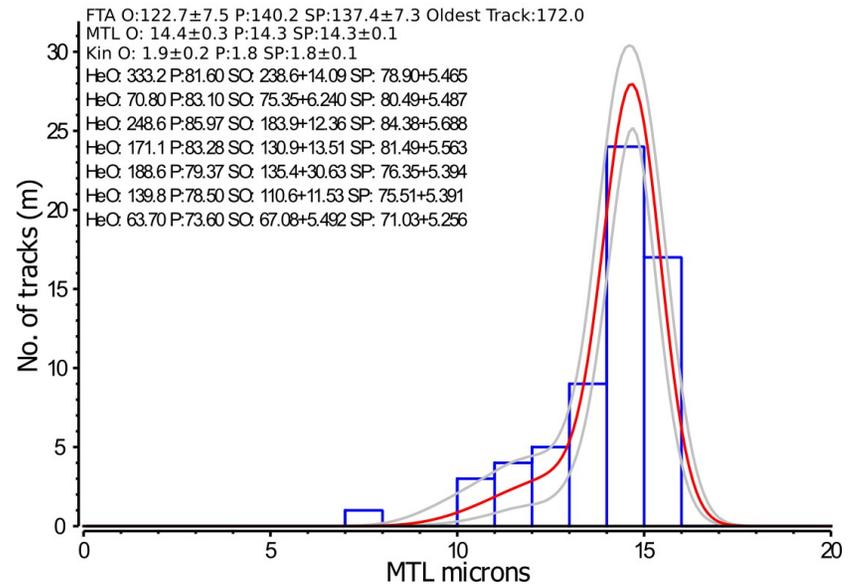
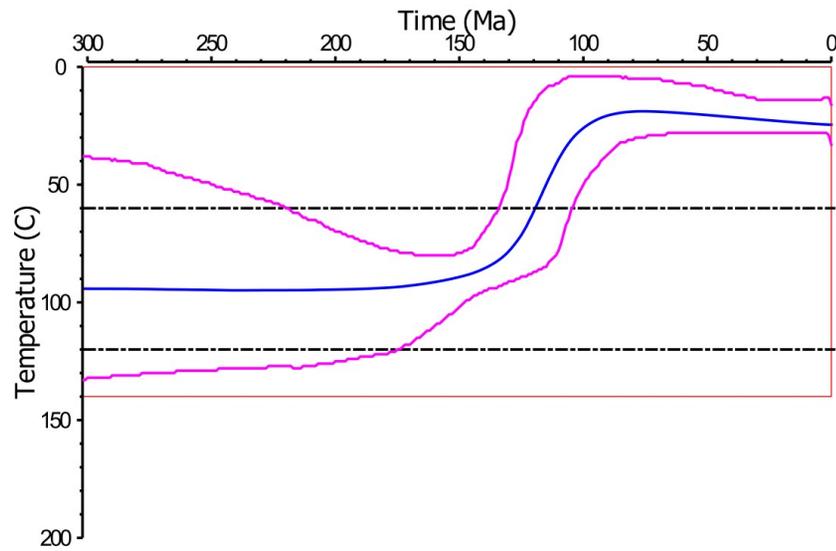
SA12-15



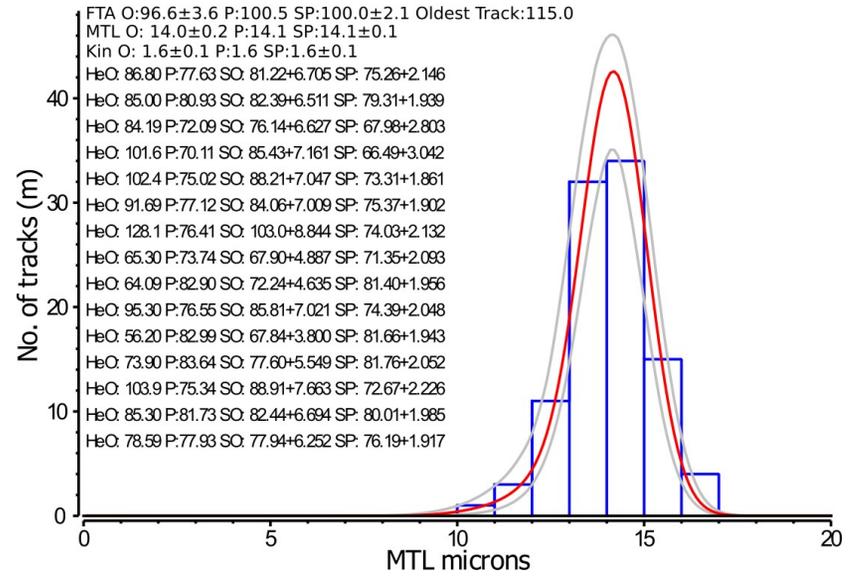
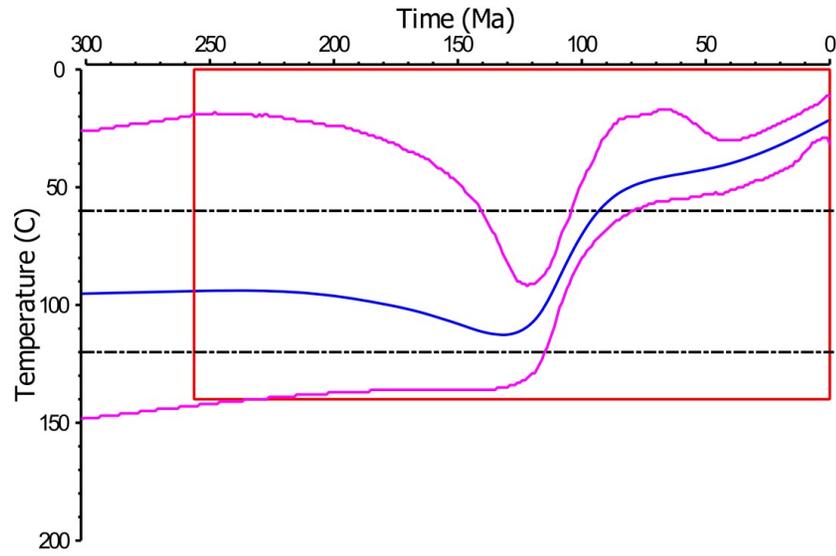
SA12-19B



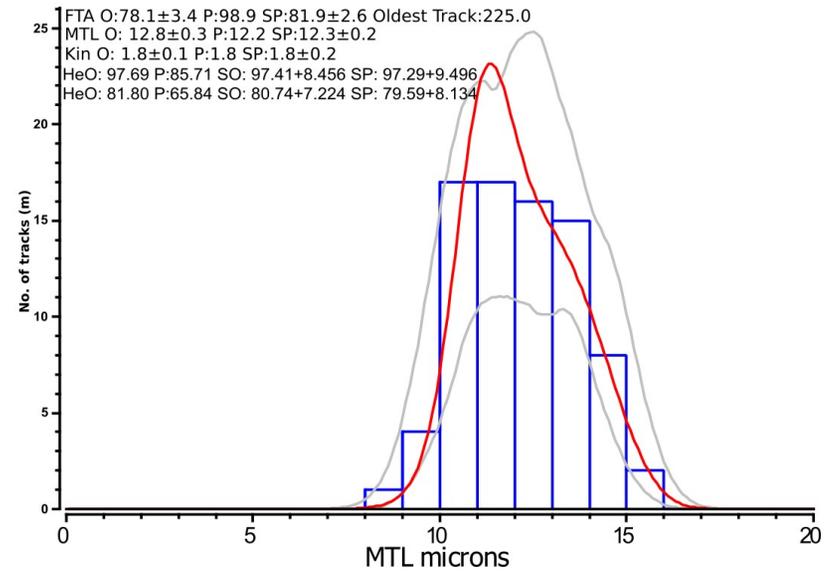
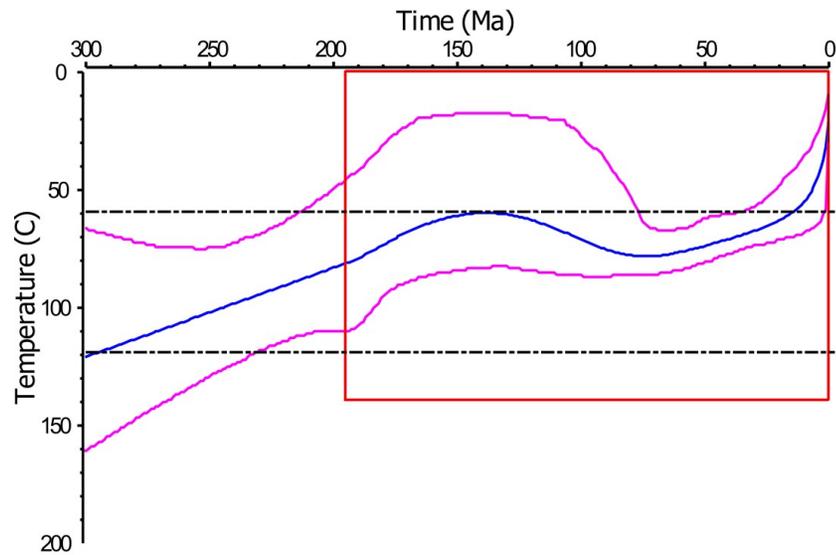
FS1605



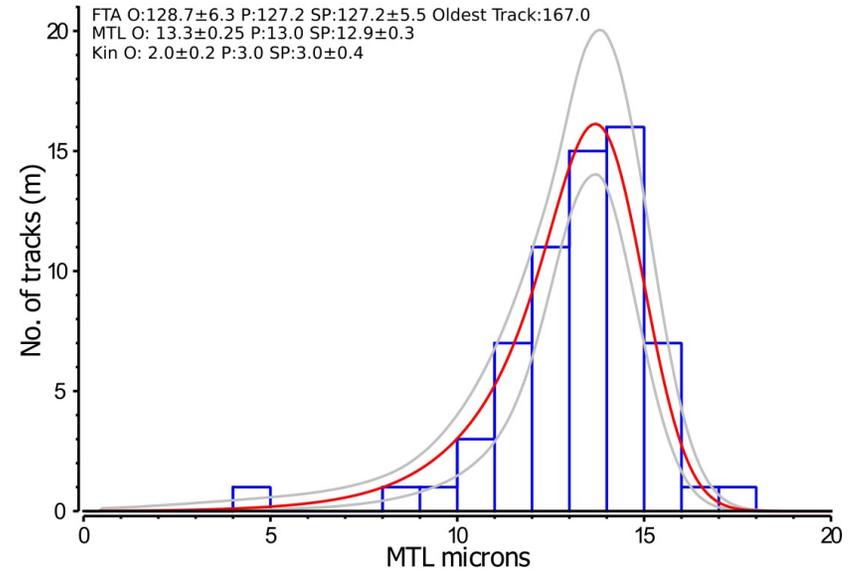
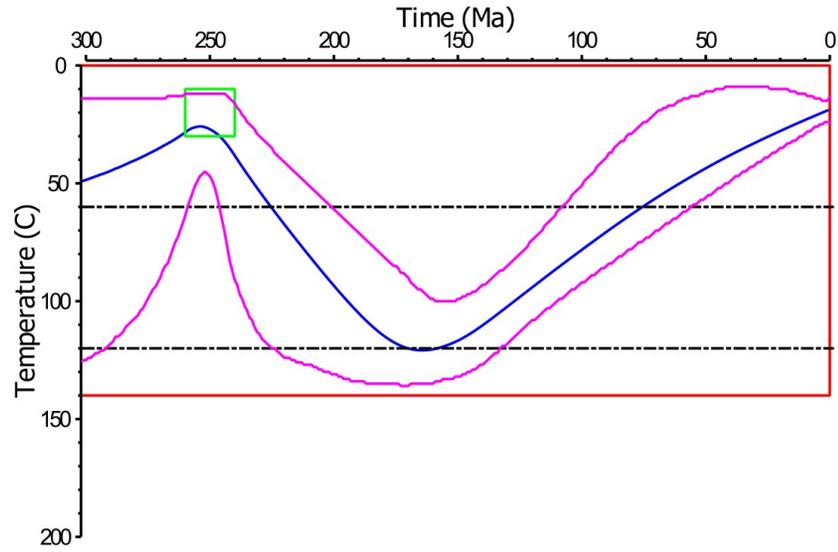
### GGO2



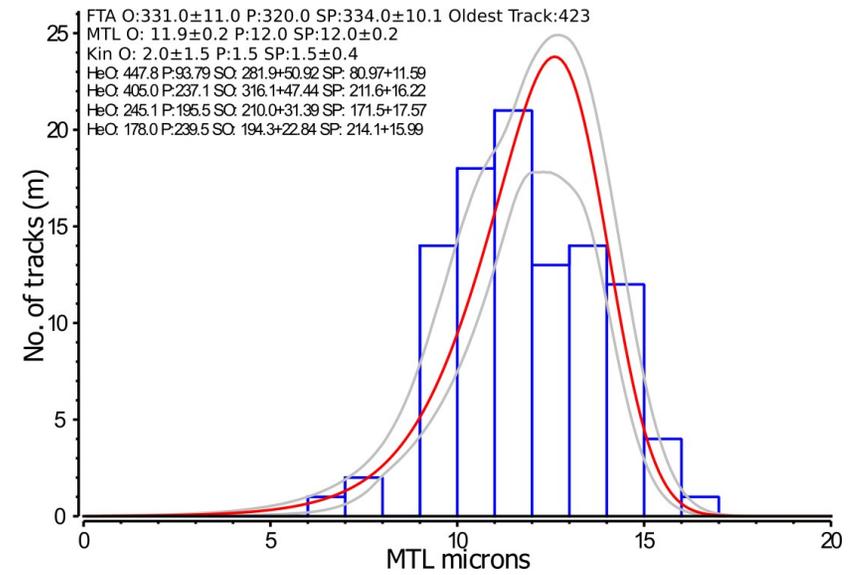
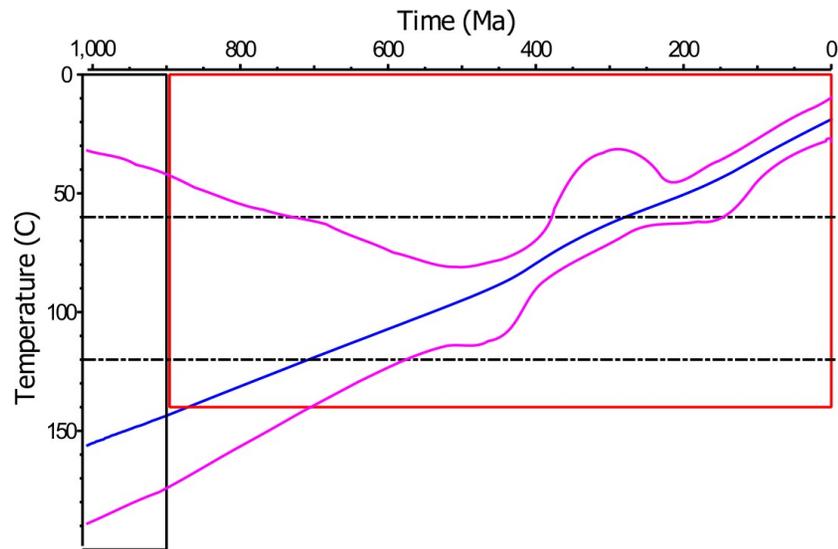
### PRU106



S-25



SR-17



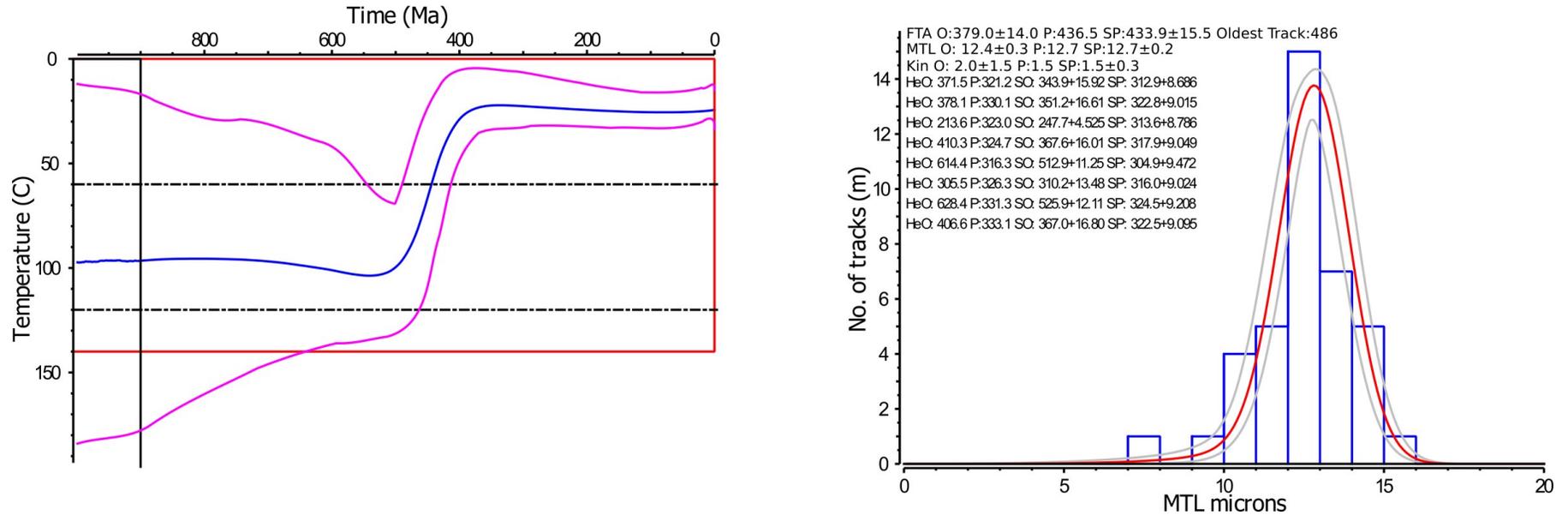
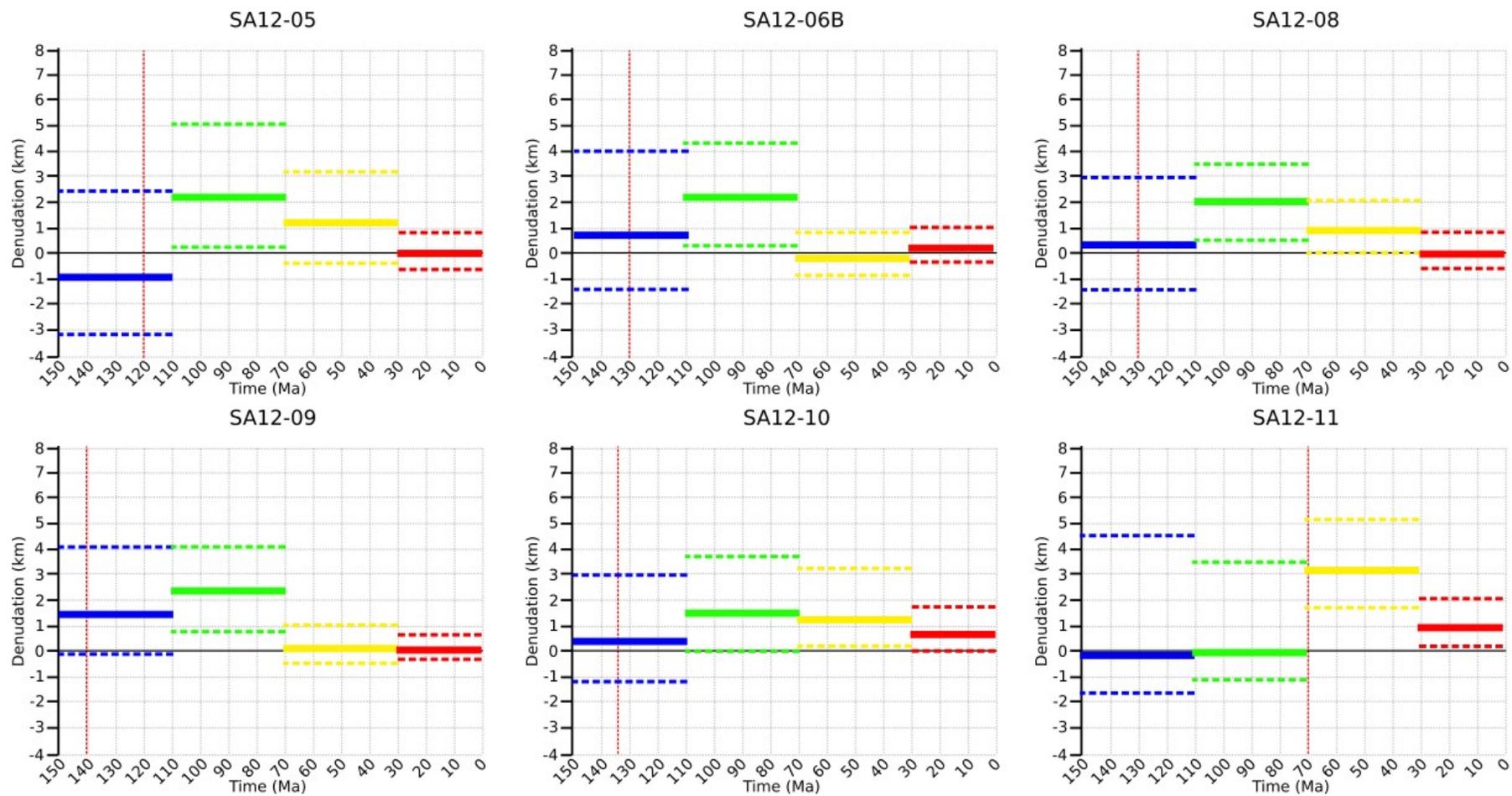
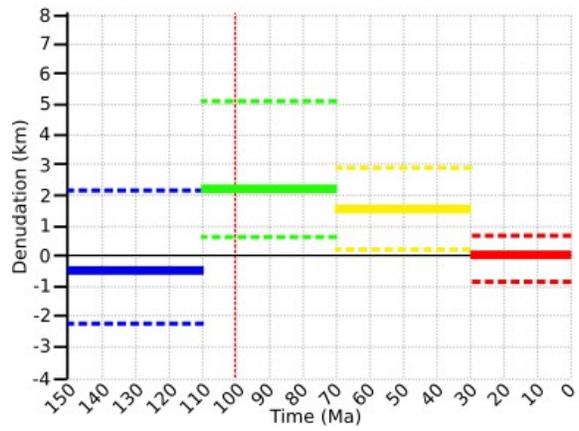


Figure S5: Additional thermal history models. Left hand figure: Blue line shows the expected model (i.e. average of all models weighted for their posterior probability); magenta lines indicate 95% credible intervals for the expected model. Red box indicates the prior information on temperature and time. Right hand figure: Histograms show the observed track-length distributions; red curve indicates the predicted track-length distributions, grey curves indicate 95% credible intervals (i.e. uncertainty) for track-length distribution prediction. FTA = fission track age, MTL = mean track length, Kin = kinetic parameter (i.e. Dpar), He = (U-Th)/He age; O = observed data, P = predicted data for the expected model, SO = sample observed (data resampled from the uncertainty range given on the observed age), SP = sample predicted (the mean and standard deviation of AHe ages for all models accepted during the inversion).

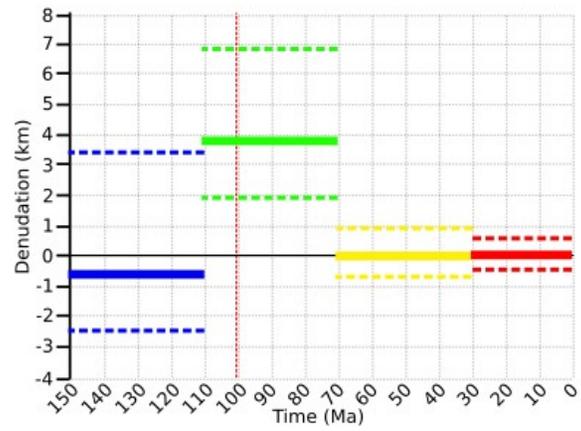
Figure S6: Denudation magnitudes.



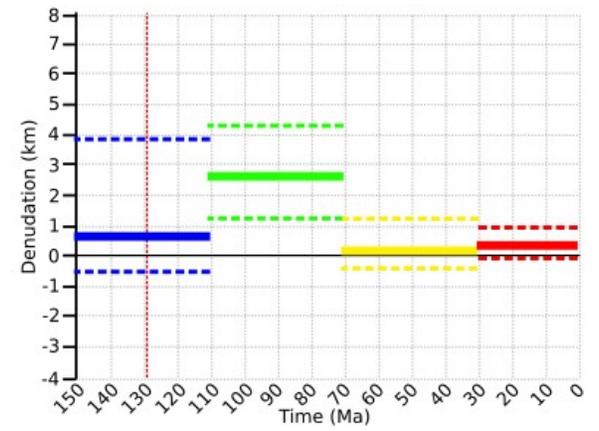
SA12-12



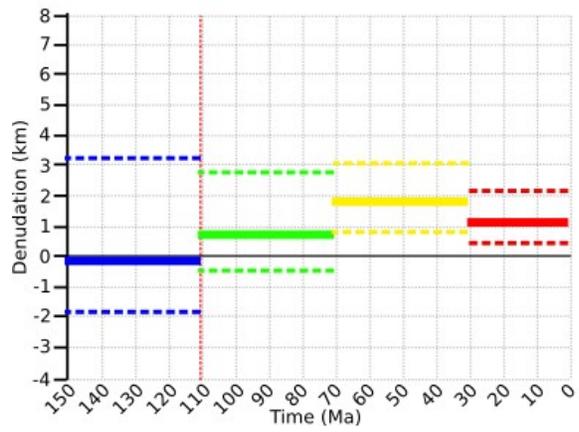
SA12-13



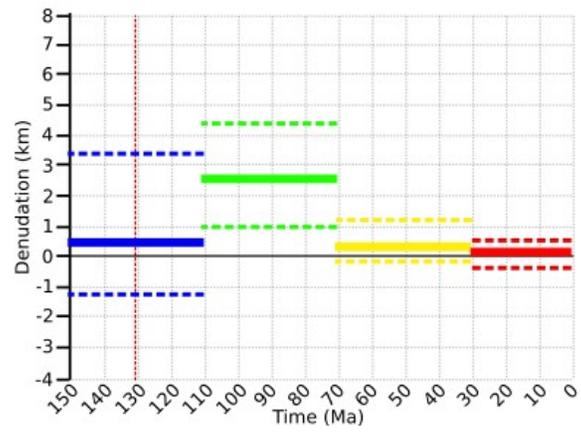
SA12-14



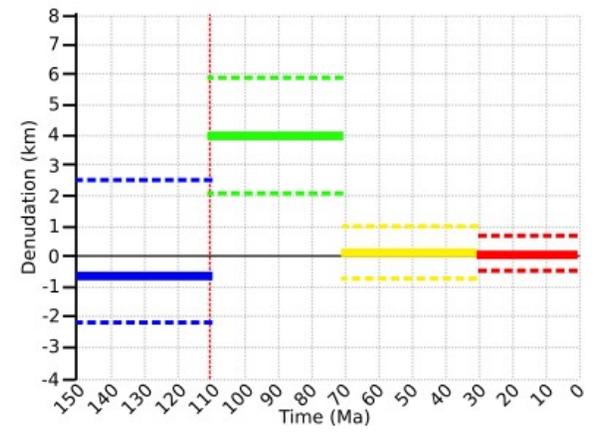
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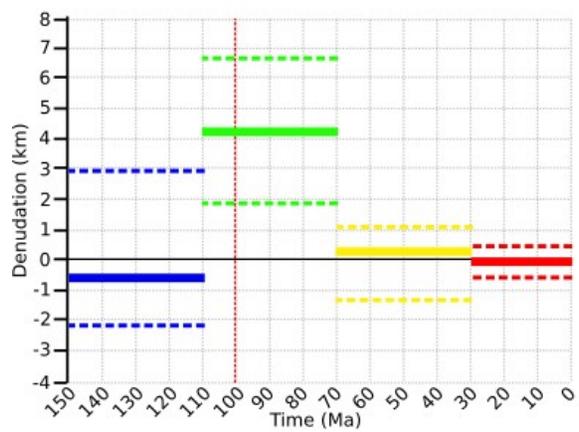
SA12-19B



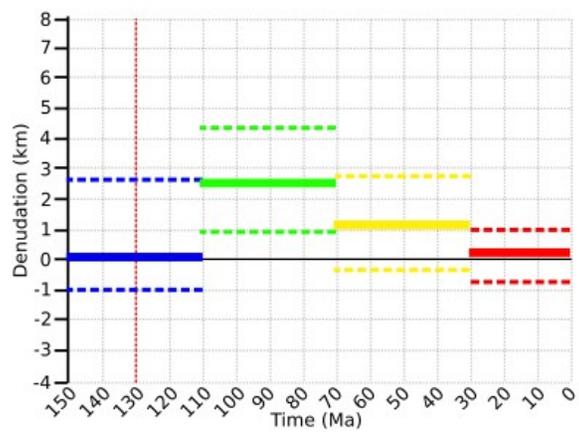
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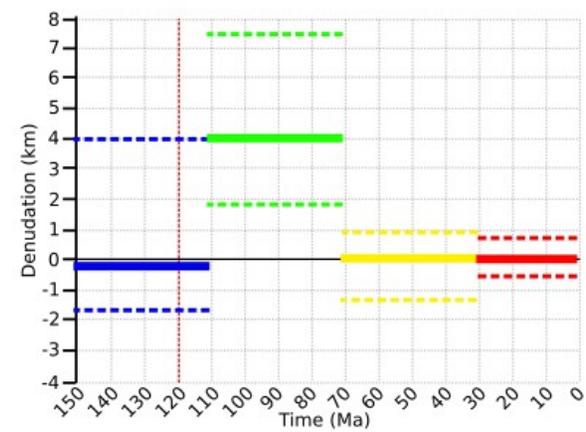
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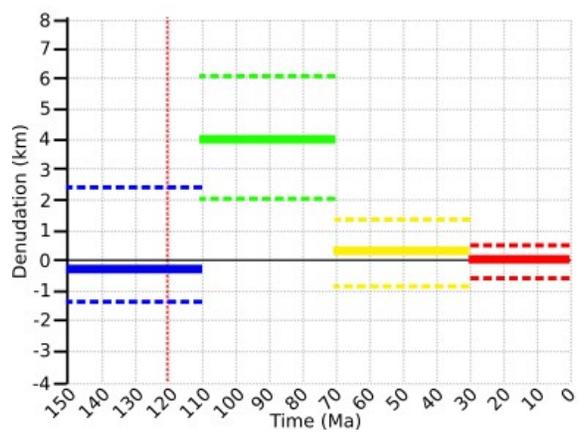
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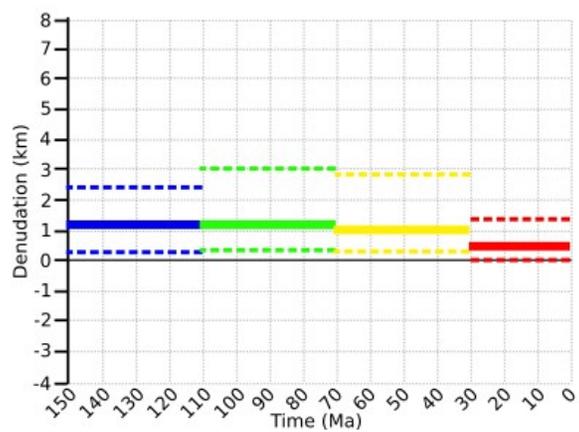
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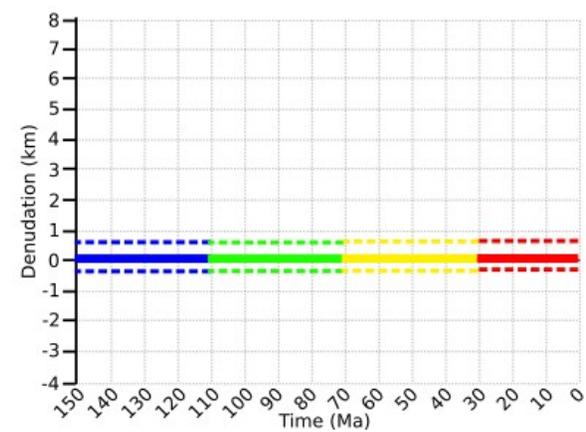
8732-88



S-25



V-10



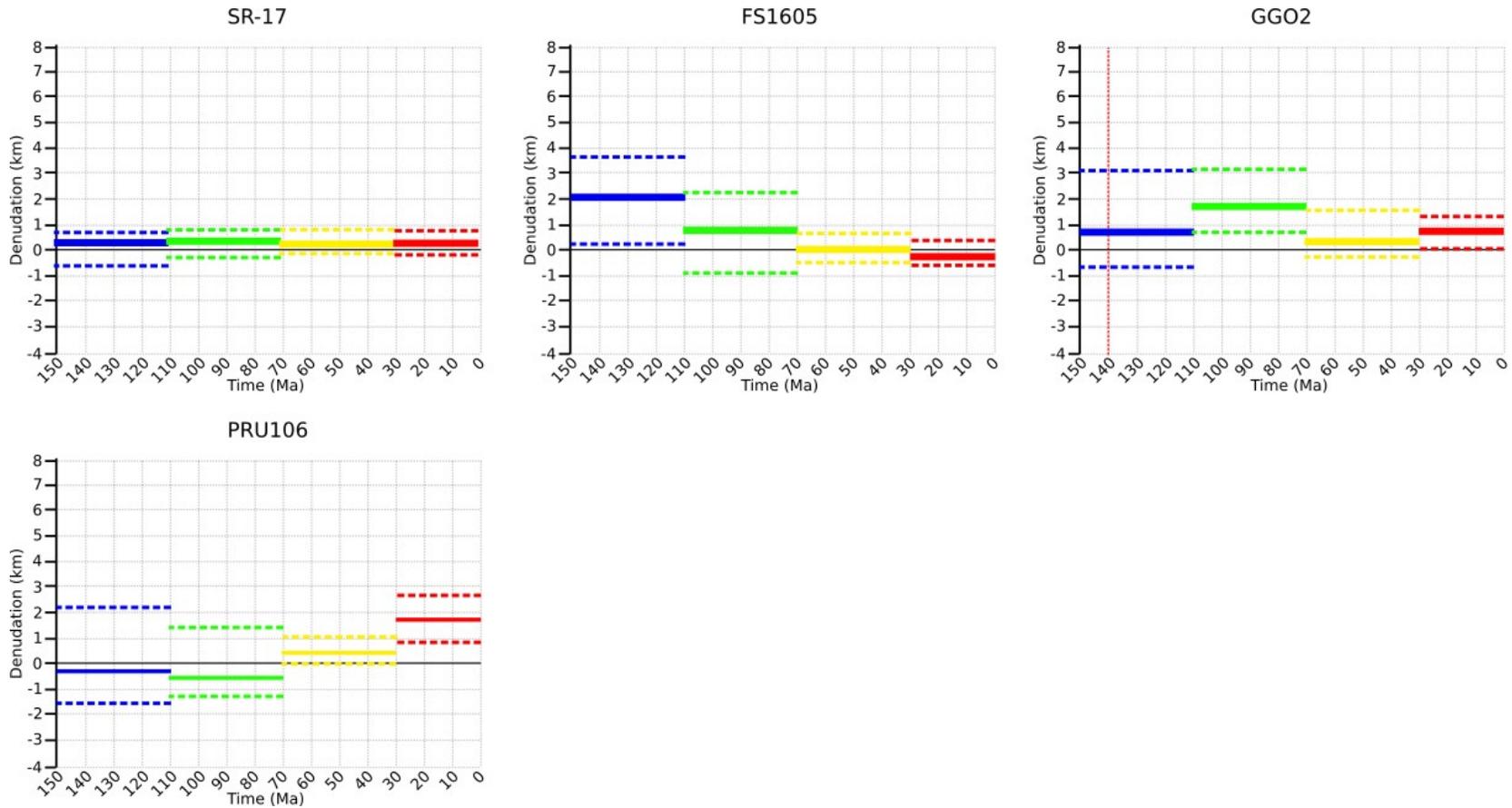


Figure S6: The expected thermal history for each sample has been used to constrain magnitudes of denudation over the intervals 150 – 110 (Blue), 110 – 70 (green), 70 – 30 (yellow) and 30 – 0 Ma (red). Denudation estimates were made by reading the change in temperature over the specified time interval from the expected thermal history model (blue t-T path in Figure S4) and dividing by an assumed geothermal gradient. In this case the geothermal gradient was assumed to be  $25 \pm 1^\circ\text{C}/\text{km}$ . The uncertainty on temperature measurements was taken as the temperature of the 95% credible intervals at the start and end time of the specified interval. The uncertainties on denudation measurements were calculated using propagation of uncertainties on the geothermal gradient and temperature estimates. However, it should be noted that the 95% credible interval range represent a probability distribution and therefore the upper and lower uncertainty levels on denudation estimates should be considered as extreme values with a much lower probability than the estimated denudation using the expected model. The red dashed line represents the time when the model indicates that the sample was last at palaeo-temperatures of 100 – 120 °C. Before this time, models are entirely unconstrained due to earlier tracks having been annealed. Because of the additional uncertainty caused by this, the early Cretaceous (150 – 110 Ma) denudation map has been removed; however, for consistency the <150 Ma denudation history for all samples has been used in these plots.

**TABLE S7**

Long. (°)	Lat. (°)	Sample Name	Elev. (m)	Rock Type	Stratigraphic Age	Lithological Unit
22.20	-29.22	FS1605	843	Granite Breccia	Proterozoic	NMP
20.30	-28.36	GGO2	846	Granite	Proterozoic	NMP
19.52	-28.48	PRU 106	783	Navos Granite	Proterozoic	NMP
23.70	-29.15	SA12-05	1007	Granite dropstone (in Dwyka tillite)	Permian	Karoo Supergroup
23.14	-29.54	SA12-06b	1068	Bt. Gneiss (in Dwyka tillite)	Permian	Karoo Supergroup
22.31	-29.52	SA12-08	1039	hbl-bt-granite-augengneiss	Proterozoic	NMP
22.12	-29.40	SA12-09	995	Qtz-fdsp-bt Gneiss	Proterozoic	NMP
21.94	-29.36	SA12-10	1075	Porphyroblastic Qtz-fdsp-bt gneiss	Proterozoic	NMP
21.94	-29.24	SA12-11	1057	Coarse grained Porphyritic Granite	Proterozoic	NMP
21.63	-29.30	SA12-12	981	Bt-Qtz-Fsdp orthogneiss	Proterozoic	NMP
21.47	-29.28	SA12-13a	962	Paragneiss	Proterozoic	NMP
21.15	-29.35	SA12-14	797	Banded pelitic gneiss	Proterozoic	NMP
20.98	-29.42	SA12-15	884	Cg. Felsic Bt-Gneiss	Proterozoic	NMP
19.53	-29.34	SA12-19b	1034	Felsic qtz-fdsp-bt gneiss	Proterozoic	NMP
26.42	-26.63	V-10	1400	Granite	Archaean	Kaapvaal craton
25.25	-27.13	SR-17	1300	Granite	Archaean	Kaapvaal craton
26.08	-29.52	S-25	1450	Felspathic-sandstone	Lower Triassic	Upper Beaufort Group

TABLE S7: Lithological details for samples.