5.0 THE SAR PROTOCOL APPLIED TO THE ROSE COTTAGE CAVE SAMPLES

5.1 Sample overview

In this chapter the OSL analyses of quartz aliquots from Rose Cottage Cave are presented. Seventeen luminescence samples, taken over the last decade, were used in this study. Fourteen of these samples were dated. In 1996 S. M Woodborne collected six luminescence samples (RCC 5-10) from the Harper excavation for part of a post-doctoral study into the MSA (Woodborne & Vogel, 1997). A total of twelve further OSL samples (RCC 11-22) were collected from RCC during 1999 and 2003 from the Wadley and Harper excavations by S. M. Woodborne and M. Pienaar. Table 5.1 presents the pretreatment affiliations of all the dated RCC samples. A total of eight of these samples were dated in Pretoria and six in Risoe, Denmark by A. S. Murray. Samples RCC 6-10 were used to characterise the OSL behaviour of samples by using two mask sizes (5mm and 2mm) and etched and un-etched quartz.

The conventional SAR protocol (Murray & Wintle, 2000) (chapter 3) was used for all the RCC samples using a preheat range between 160ºC and 300ºC for 10 s with a cut-heat of 160ºC for 0s. The L_x and T_x measurements were acquired through a range of regenerative OSL measurements each stimulated at 125ºC for 40 s with blue LEDs at 90% power. All measurements were carried out on an automated Risø TL/OSL DA 15 reader with a measured systematic error of 1.8%. Behavioural controls inherent to the conventional SAR protocol were administered to each sample and depositional factors such as partial bleaching were assessed using signal analysis methods (Bailey, 2003a, 2003b). The grain size was chosen based on the distribution of isolated quartz extracts.

Table 5. 1 RCC samples in depth order showing grain size fractions and mask sizes used in dating

RCC OSL SAMPLES		
Sample name	***Size Fraction***	***Mask size***
RCC 17		
RCC 22		
RCC 21	180-212µm	2mm
RCC 20		
RCC 10	180-212µm	5mm & 2mm
RCC 19	180-212µm	2mm
RCC 9	180-212µm	5mm & 2mm
RCC 18	180-212µm	2mm
RCC 16	180-212µm	Measured at Risoe
RCC 6	106-150µm	5mm & 2mm
RCC 15	180-212µm	Measured at Risoe
RCC 14	180-212µm	Measured at Risoe
RCC 7	180-212µm	5mm & 2mm
RCC 13	180-212µm	Measured at Risoe
RCC 12	180-212µm	Measured at Risoe
RCC 8	106-150µm	5mm & 2mm
RCC 11	180-212µm	Measured at Risoe

5.2 Rejection criteria

The aliquots were assessed against OSL rejection criteria that were assumed to eliminate most errors with the OSL behaviour and sedimentary history of the grains. The following rejection tests were applied in order to obtain correct D_e values:

- A dose recovery experiment
- A partial bleach test
- A feldspar elimination test (IR-OSL depletion ratio)
- Sensitivy correction in regeneration analysis
- A recycling ratio Test (RR)
- A test for recuperation (charge transfer), and
- Must present a preheat plateau.

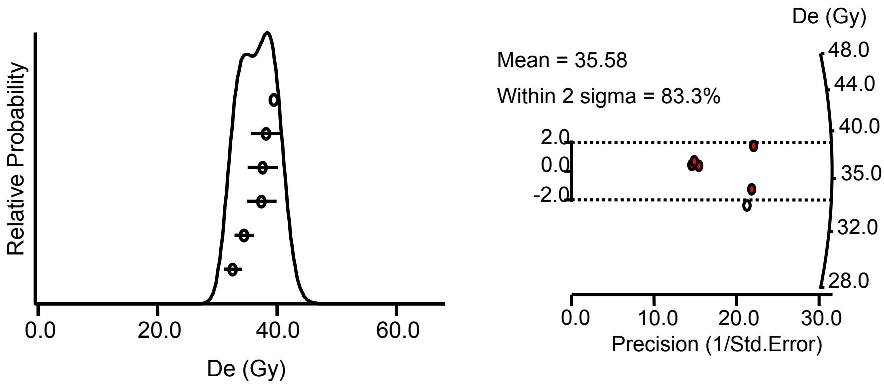
5.2.1 Dose recovery experiment

Dose recovery experiments (Murray & Roberts, 1998; Roberts *et al.*, 1999) were performed on six aliquots per sample. The aliquots were optically bleached using blue LEDs for 40s at 125°C. After zeroing, a known laboratory dose assumed to be representative of the natural dose, was administered and a conventional SAR protocol was performed to see if the dose could be recovered. The test is an independent verification of the SAR protocol and any sensitivity changes that could occur from the natural OSL signal are detected. The test does not re-create time-dependent effects that occur naturally and could therefore result in slight differences between the natural dose and the recovered dose. The test does, however, give a good indication of the reproducibility of the D_e value and the appropriateness of using sensitivity corrected dose response curves throughout a SAR measurement sequence. Samples RCC 6-10 and RCC 18 were used as general indicators for all the Rose Cottage Cave material in this experiment. Table 5.2 shows the results of dose recovery experiments, where the average recovered dose vs. the given dose is quoted. All values recovered well and it was assumed that all RCC samples have little if any sensitivity changes in the natural OSL measurement. Graphical displays in the form of radial plots and relative probability plots are shown in figure 5.1

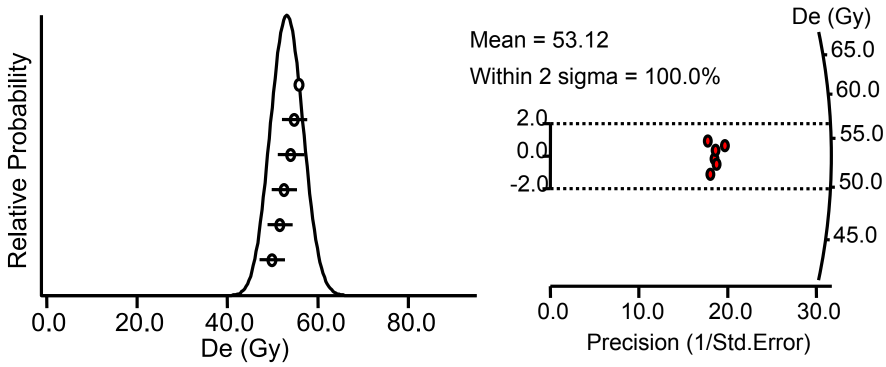
Table 5. 2 Values of recovery doses obtained by SAR protocol for known doses using a dose recovery experiment (Murray & Roberts, 1998; Roberts *et al*., 1999). Samples are presented in order of decreasing depth.

Sample Name	Administered Dose	Recovered Dose	% Dose Recovery
RCC 10	35.8 Gy	35.6 Gy	97.8%
RCC 9	53.8 Gy	53.1 Gy	98.7%
RCC 18	53.8 Gy	53.8 Gy	100.0%
RCC 6	89.6 Gy	89.1 Gy	99.4%
RCC 7	134.4 Gy	134.5 Gy	99.9%
RCC 8	134.4 Gy	123.6 Gy	92.0%

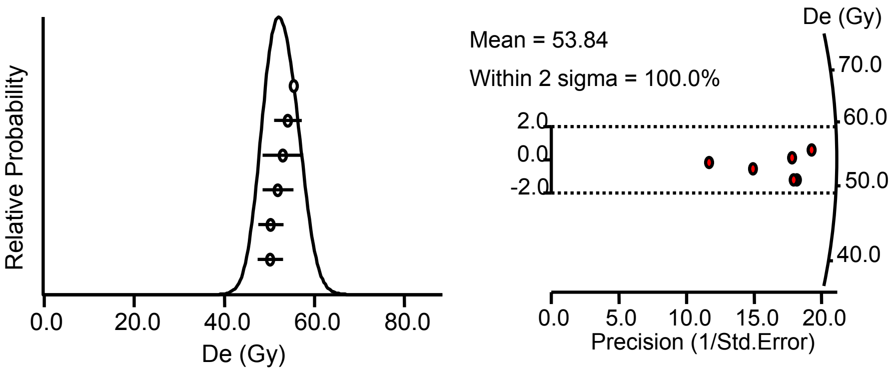
RCC 10



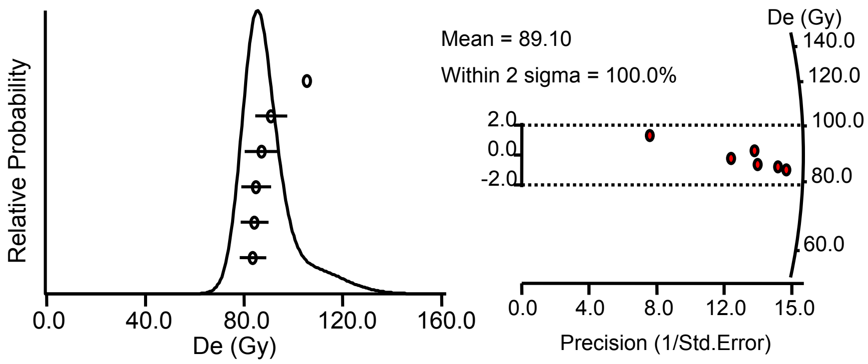
RCC 9



RCC 18



RCC 6



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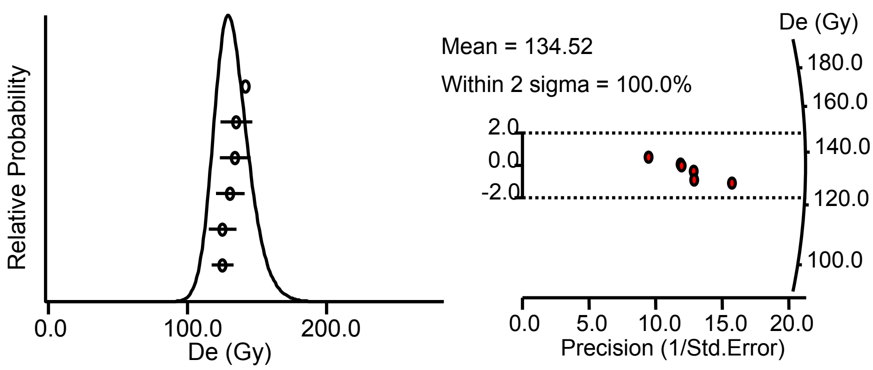


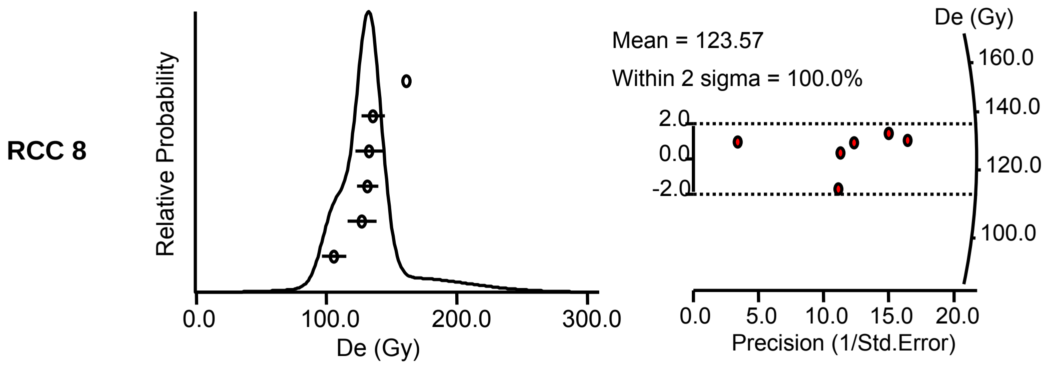


Figure 5. 1 Values of recovery doses obtained by SAR protocol for known doses using a dose recovery experiment (Murray & Roberts, 1998; Roberts *et al*., 1999). Samples are presented in order of decreasing depth.

5.2.2 Partial bleach test

Both Butzer (1984a, 1984b) and Herries & Latham (2002) have suggested that the deposit in Rose Cottage Cave derives from material detached from the cave roof and walls, introduced through a major joint intersection at the back of the cave that was enlarged by water erosion. Butzer suggested that the deposition of the cave consists of predominantly weathered sandstone klasts, produced through weathering or rock spalling of the cave roof and walls and laminated sediments brought in by water. The possibility of incomplete signal resetting needs to be assessed. In this study signal analysis methods (discussed in chapter 2) were used to determine incomplete signal resetting (Bailey, 2003a, 2003b). The $D_e(t)$ method of Bailey was performed on samples RCC 6-10, 18, 19 and 21. Ten random aliquots representative of a preheat range between 160°C and 300°C were used for each sample.

The method requires that the OSL signal comprise a thermally stable fast bleaching and a slow bleaching component. If the sample is partially bleached then a slower bleaching components would yield a greater residual signal i.e. yield a greater age. Stimulation was carried out at 125°C for 40s using blue LEDs at 90% power. Values of D_e were obtained for each aliquot using approximately 0.5 second integration channels and are shown in stratagraphic order in figure 5.2. The downward right trend in figure 5.2 indicates that the fast component could not have been preferentially bleached. This is based on the assumption that if partial bleaching occurs, the fast component would bleach first and there would be a higher residual signal from an integration channel in the medium component. This is not characteristic of fluvial or spalled material and could indicate aeolian deposition.

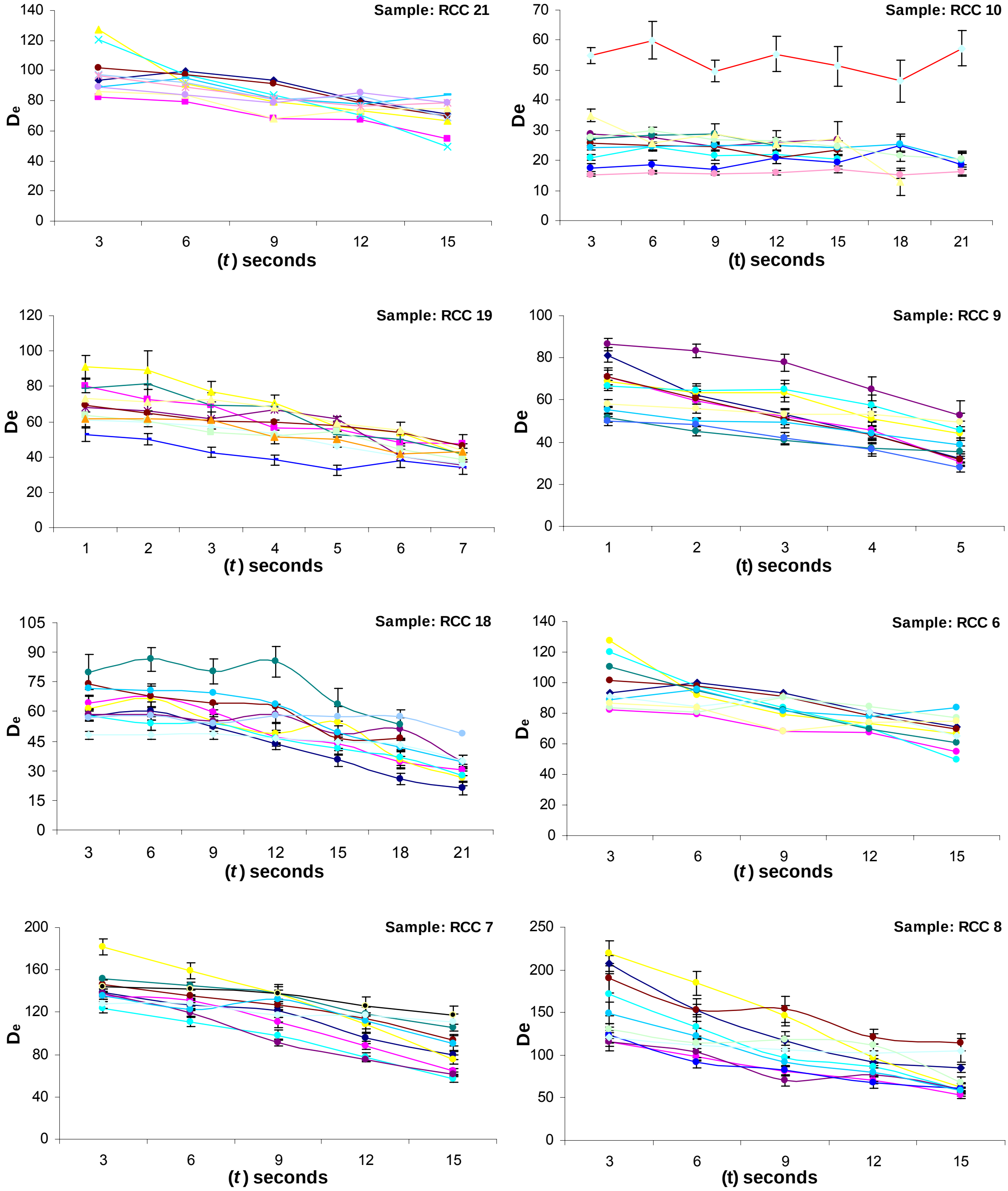


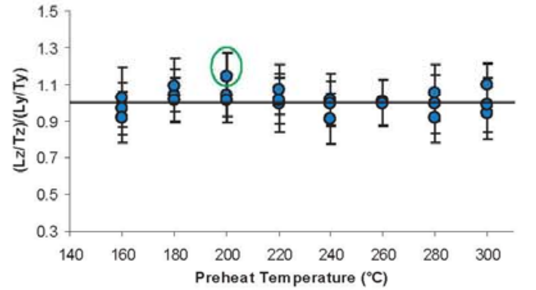
Figure 5. 2 D_e (*t*) plots displaying D_e values quoted in Gy obtained at different integration channels (Bailey, 2003a).

5.2.3 IR-OSL depletion ratio test

Feldspar contamination in quartz aliquots usually results in higher D_e value estimates in OSL measurements. This is due to an internal potassium contribution that increases the internal dose but is not considered in the dosimetry or is the result of highly sensitive feldspars that have not been eradicated through pre-treatment techniques. If random trap emptying synonymous with feldspars, also known as anomalous fading, occurs (Aitken, 1998), then feldspar contamination could also result in an underestimation of D_e. It is therefore important to assess whether scatter in the equivalent dose measurements is a result of feldspar contamination. The IR OSL depletion ratio test consists of 3 measurement cycles. The first measurement (L_1/T_1) consists of a typical regenerative dose, usually the first regenerative point. The second measurement (L_2/T_2) is a repeat of the first measurement. Typically the ratio between these two measurements would fall within unity if no other measurement is applied. Before the second measurement however, the sample is stimulated with IR diodes. IR stimulation at approximately 880 nm does not significantly affect quartz (Stokes, 1992; Duller, 2003) and any depletion of the signal measured during L_2/T_2 would be the result of feldspar contamination.

Figure 5.3 shows the OSL IR depletion ratio test using large (5mm) mask sizes, 2mm mask sizes and the results of samples that were etched in HF acid. The initial dose was 18 Gy measured with blue LEDs for 40s. A repeat dose of 18 Gy was stimulated using IR diodes at 90% power for 100s followed by stimulation from blue LEDs for 40s. Where the depletion ratio deviated from normality by more than 2σ, calculated from the measurement precision, the aliquots were rejected from further analysis (Jacobs *et al.,* 2003a). Figure 5.3 shows the results with two standard deviation error bars. The red circles indicate aliquots that were rejected due to feldspar contamination; the green circles represent aliquots that were rejected due to unexplained recycling ratio behaviour.

Out of a total of 408 aliquots, 101 or 25% of the aliquots were rejected due to feldspar contamination. If the etched material is not included then a total of 98 out of 240 aliquots (41%) of the aliquots would have been rejected. This implies that in a feldspar rich site such as RCC not all feldspars are removed by mineral separation, or there is the possibility of internal feldspar inclusions. It is therefore crucial that the samples are etched in HF. The accepted samples, except for the 5mm mask sized samples RCC 10, 6, and 7 however, statistically constitute a large enough population to accept further analyses.

<table>
<tr><td></td><td>5mm Mask size
(un-etched material)</td><td>2mm Mask size
(un-etched material)</td><td>2mm Mask size
(etched material)</td></tr>
<tr><td>RCC Sample 21
180-212 µm</td><td></td><td></td><td></td></tr>
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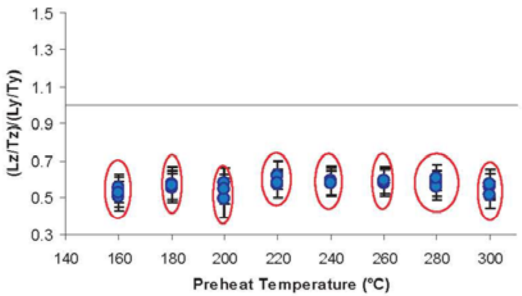
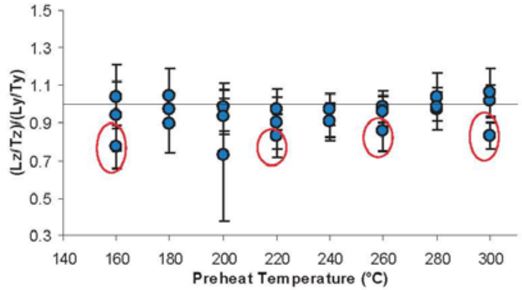
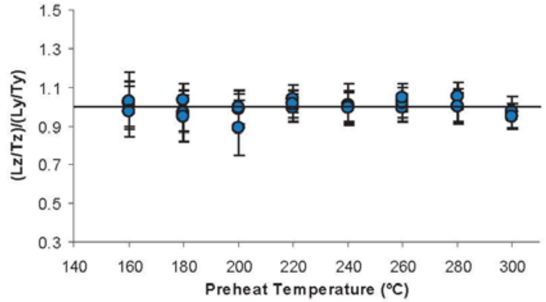
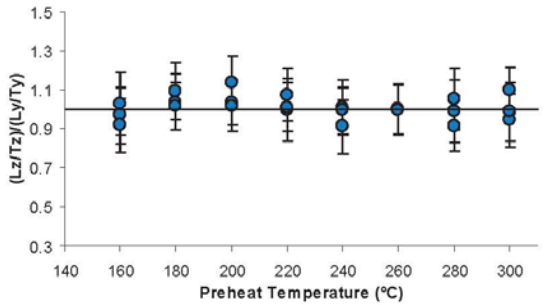
180-212 µm</td><td></td><td></td><td></td></tr>
<tr><td>RCC Sample 19
180-212 µm</td><td></td><td></td><td></td></tr>
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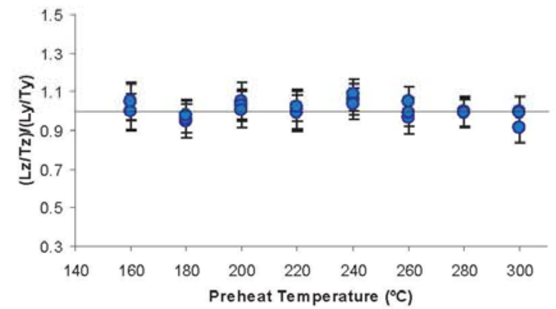
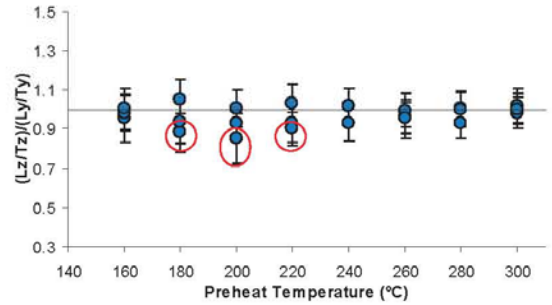
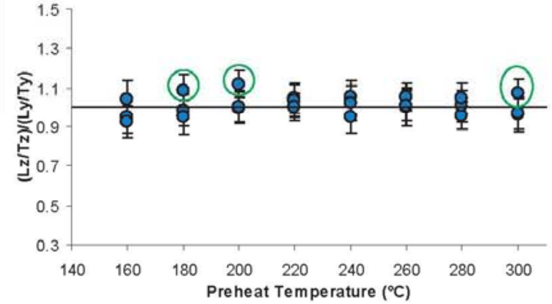
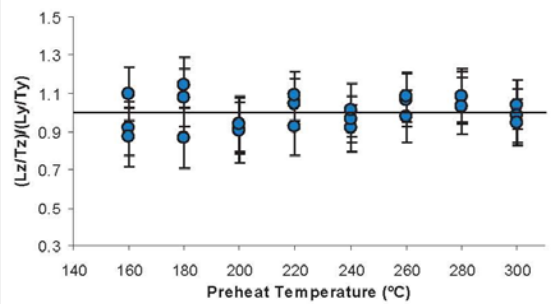
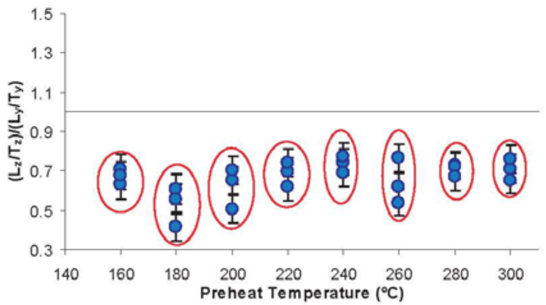
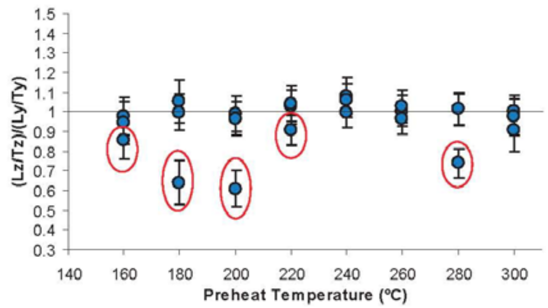
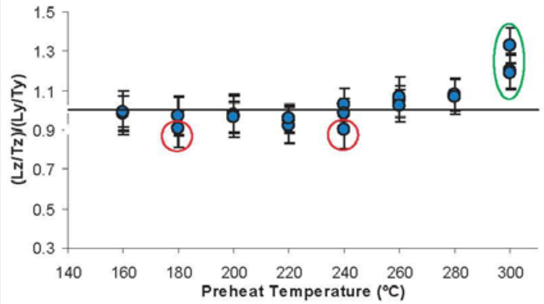
	5mm Mask size (un-etched material)	2mm Mask size (un-etched material)	2mm Mask size (etched material)
RCC Sample 9 180-212 µm			
RCC Sample 18 180-212 µm			
RCC Sample 6 106-150 µm			

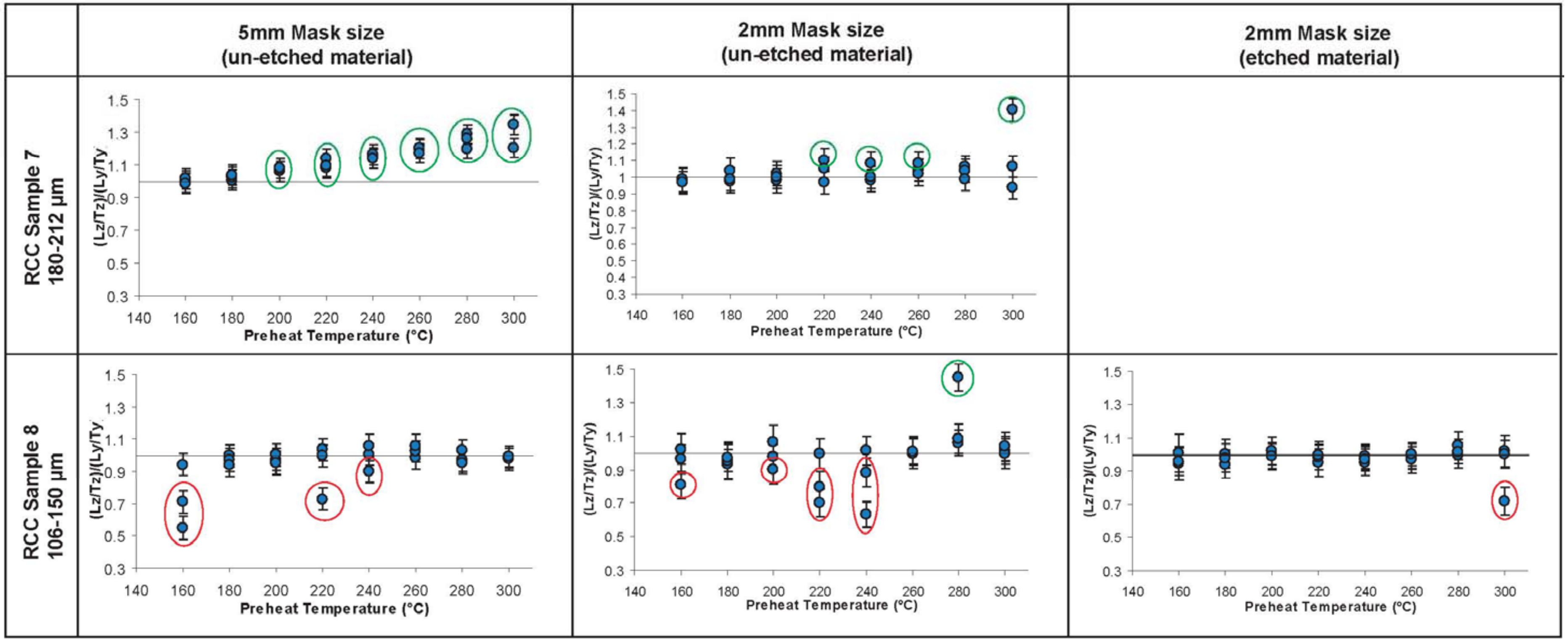
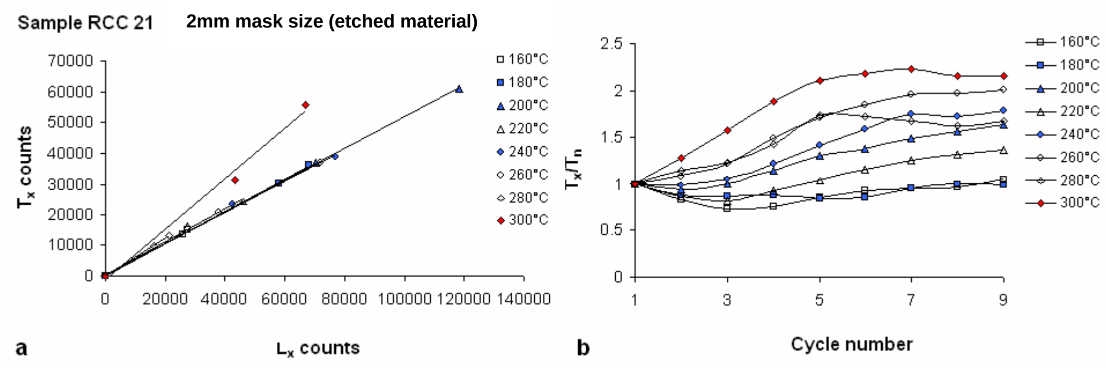


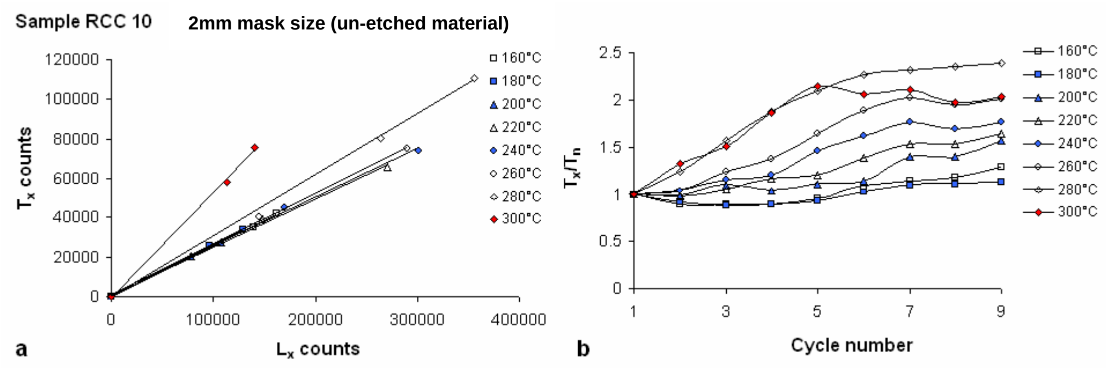
Figure 5. 3 IR OSL Depletion Ratio tests performed on aliquots using 5mm and 2mm mask sizes at different preheat temperatures. The IR OSL Ratio is given as $[(L_z/T_z)/(L_y/T_y)]$, were L and T are the regenerated OSL intensity of the given dose and the test dose. Large mask size samples RCC 6, 7 and 10 were completely rejected as measurement ratios deviated substantially between recycled points, resulting in a very low ratio. The reduction in mask size from 5mm to 2mm shows the effect of feldspar elimination. On average 20% of the aliquots were rejected from 2mm mask size samples. Samples that were etched have an average of 7% rejections.

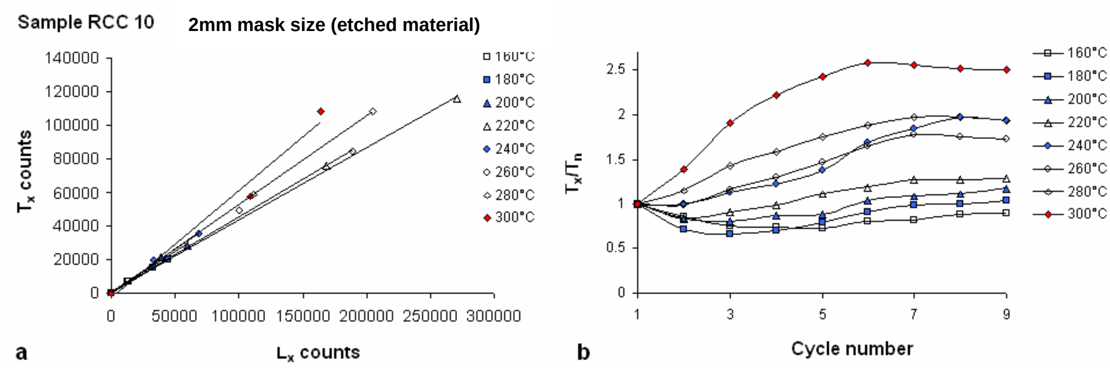
5.2.4 Sensitivity change

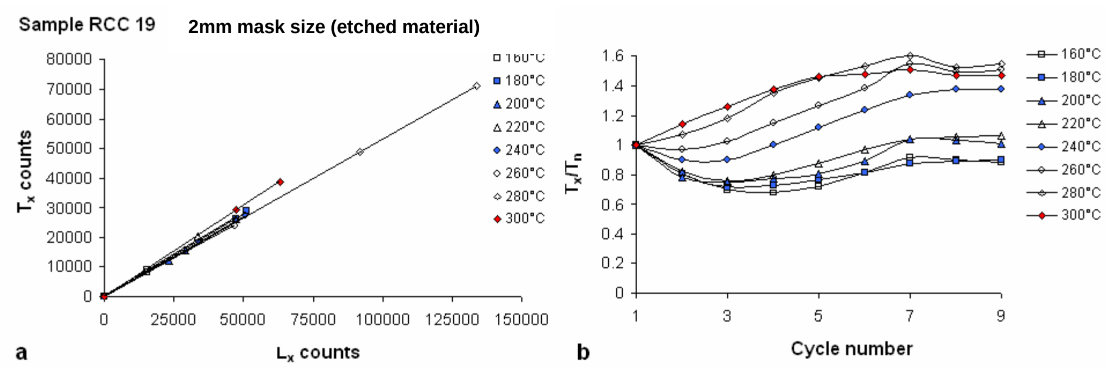
In this test a small test dose (roughly 10% of the natural D_e) is administered into a measurement sequence (Murray & Roberts, 1998; Wintle & Murray, 1999; Murray & Wintle, 2000). The test dose is used to normalize a L_x measurement. Any sensitivity changes relate to the transfer of charge produced by laboratory irradiation and preheating. Sensitivity changes can be observed as a function of preheating as well as measurement cycle. The use of a preheat range between 160°C and 300°C is used (Murray *et al.,* 1997) in order to isolate a signal derived from thermally stable traps and to equalize sensitivity between the natural and laboratory irradiated measurements. Murray & Mejdahl (1999) demonstrated that the relationship between the OSL test dose and an OSL regenerative dose must be linear for sensitivity to be corrected.

The linear relationship between L_x and T_x is shown in figure 5.2 using large and small mask sizes and a range of preheat temperatures between 160°C and 300°C. The values of L_x are plotted against T_x for two regenerative dose cycles, the values of T_x are then normalized to T_n (the test dose administered after measurement of the natural) and show the sensitisation process that occurs during a typical SAR measurement cycle. There is an initial decrease in sensitivity for the lower preheat temperatures (160°C - 240°C) and an initial increase in sensitivy for the higher preheat temperature (260°C - 300°C). If the values increase and decrease by about 25% to 35% they demonstrate typical behaviour (Jacobs *et al.,* 2003a) and it can be assumed that sensitivity changes are corrected successfully. Note only samples that have been accepted after the feldspar contamination test are shown in figure 5.4, and that all the samples show typical OSL quartz behaviour.

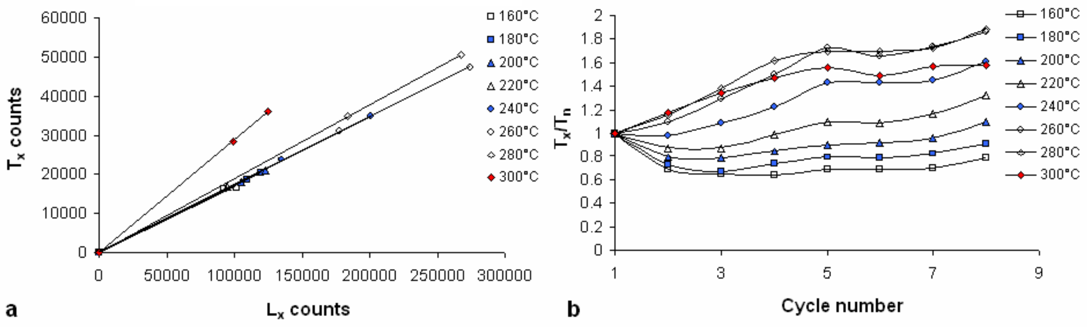

Sample RCC 21
2mm mask size (etched material)
T_x counts
L_x counts
160°C
180°C
200°C
220°C
240°C
260°C
280°C
300°C
T_x/T_n
Cycle number
a
b


Sample RCC 10
2mm mask size (un-etched material)
T_x counts
L_x counts
160°C
180°C
200°C
220°C
240°C
260°C
280°C
300°C
T_x/T_n
Cycle number
a
b

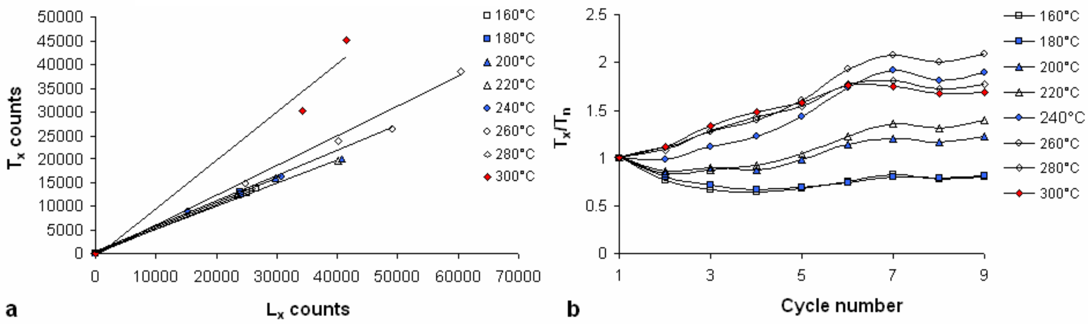

Sample RCC 10
2mm mask size (etched material)
T_x counts
L_x counts
160°C
180°C
200°C
220°C
240°C
260°C
280°C
300°C
T_x/T_n
Cycle number
a
b


Sample RCC 19
2mm mask size (etched material)
T_x counts
L_x counts
160°C
180°C
200°C
220°C
240°C
260°C
280°C
300°C
T_x/T_n
Cycle number
a
b

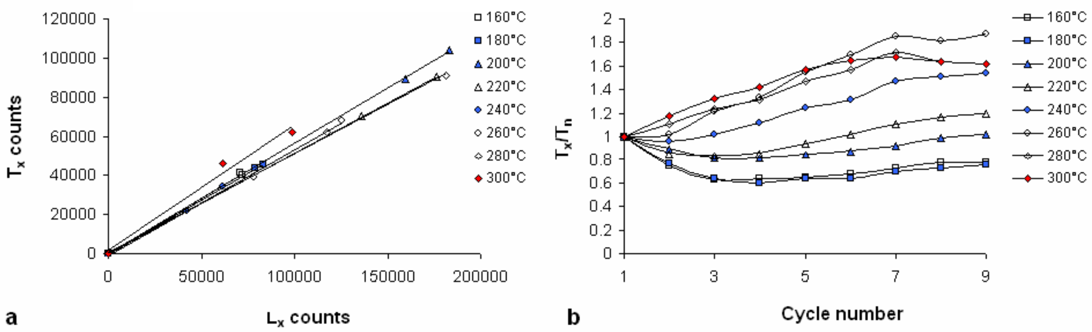
Sample RCC 9 5mm mask size (un-etched material)



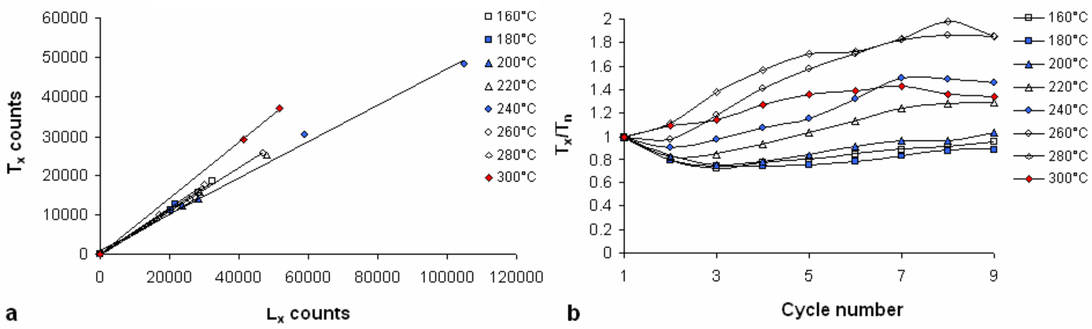
Sample RCC 9 2mm mask size (un-etched material)



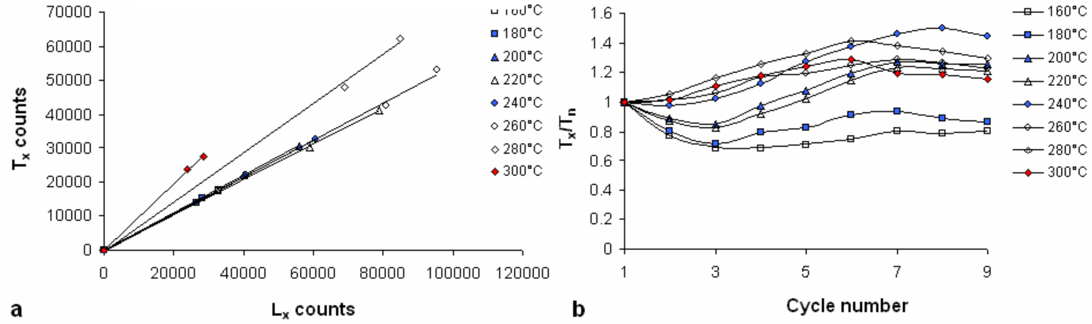
Sample RCC 9 2mm mask size (etched material)



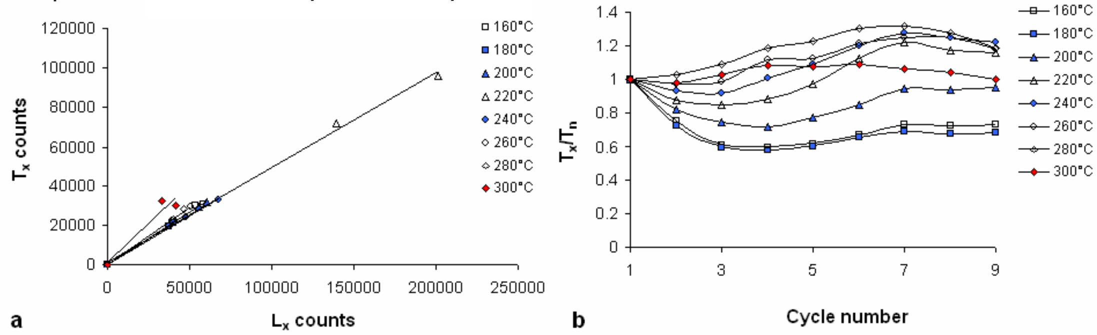
Sample RCC 18 2mm mask size (etched material)



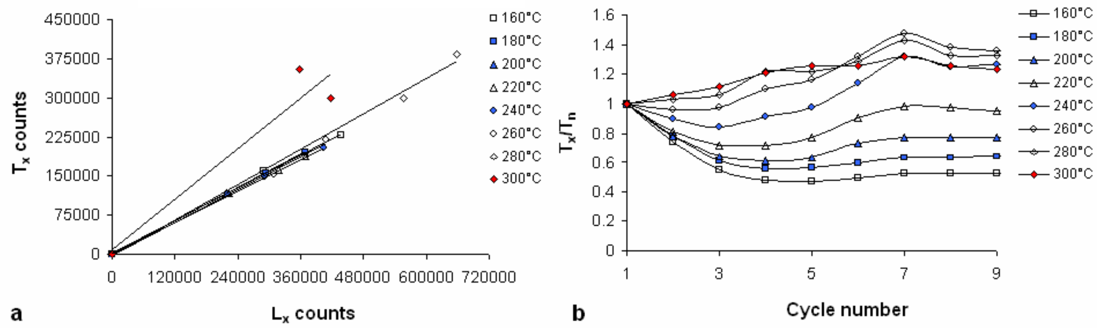
Sample RCC 6 2mm mask size (un-etched material)



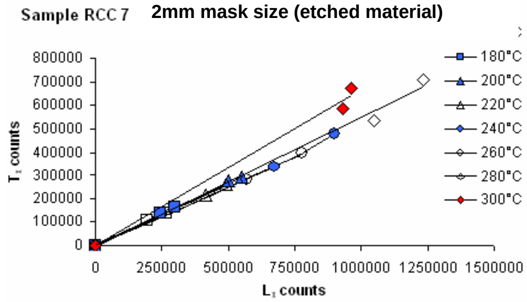
Sample RCC 6 2mm mask size (etched material)

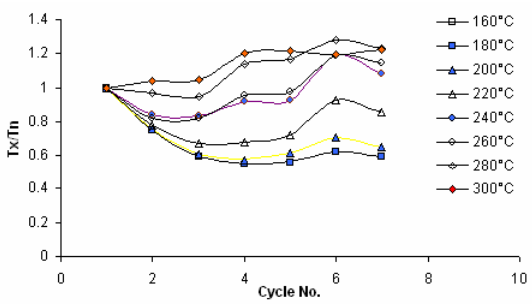


Sample RCC 7 2mm mask size (un-etched material)



Sample RCC 7 2mm mask size (etched material)





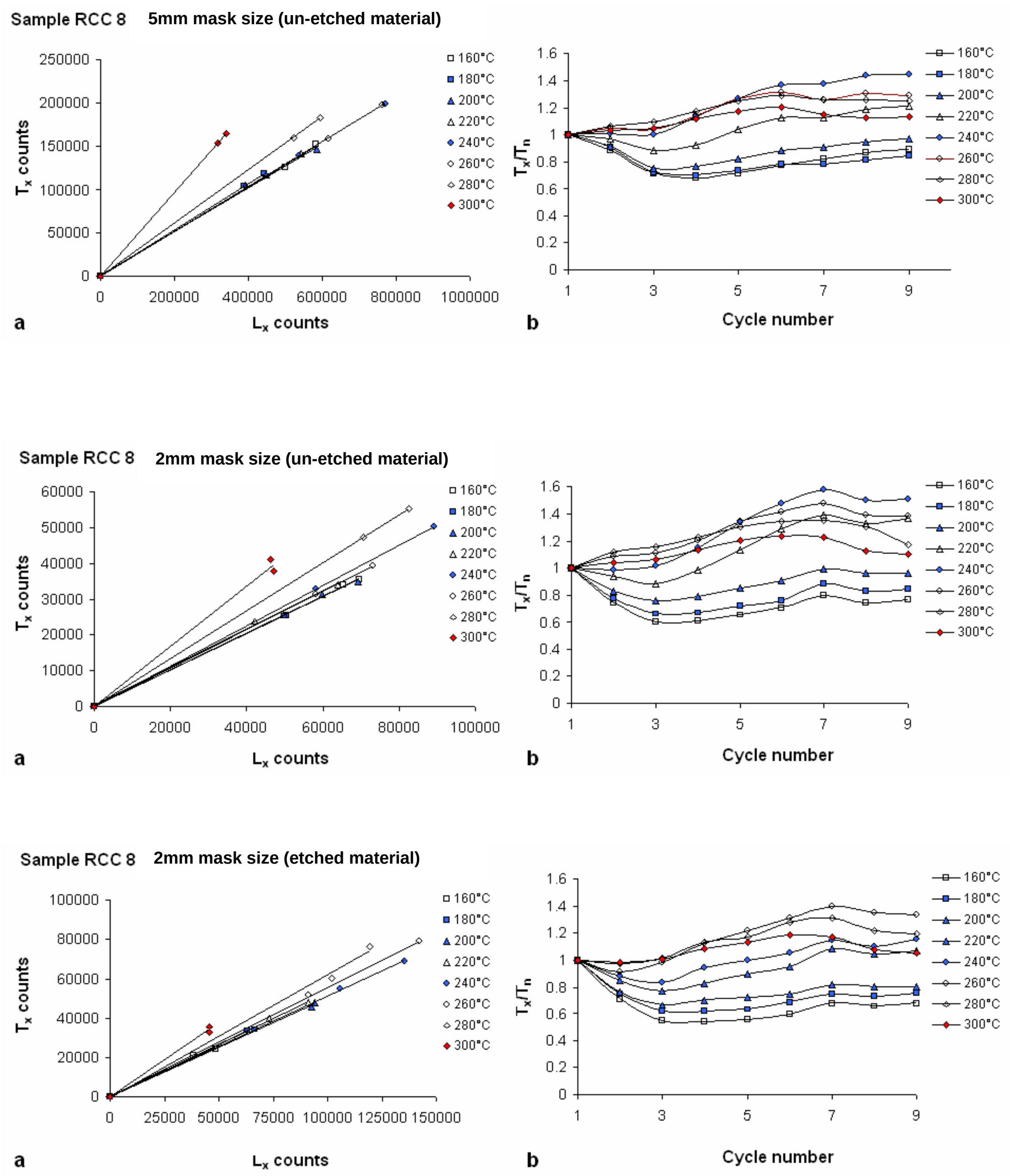
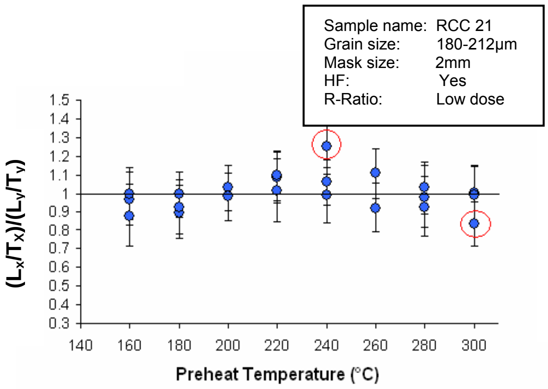
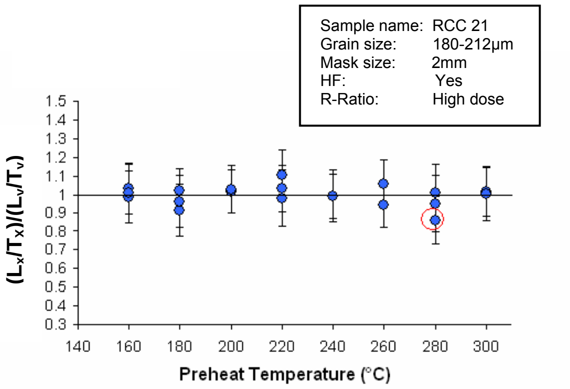


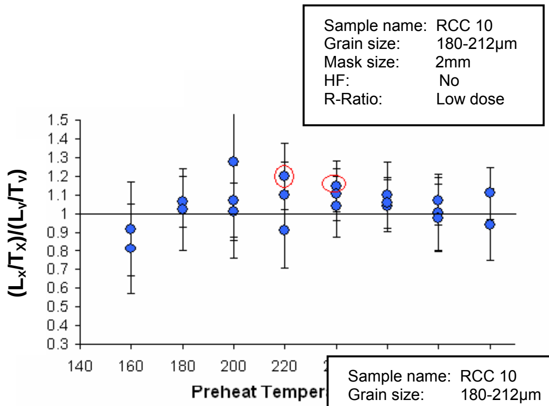
Figure 5. 4 Sensitivity changes monitored by the relationship between L_x/T_x and T_x/T_n vs. SAR cycle number. The checks are performed on aliquots using large (5mm) and small (2mm) mask sizes. The repeated dose points representing L_X/T_x values were obtained through recycling low dose data points. The 300°C values generally deviate from the main trend due to thermal depletion of the L_x measurement. The values of T_x are normalized to $T_{n,}$ the lower preheat temperatures show a decrease in sensitivity for the first few measurement cycles. The higher preheat temperatures have an increase in sensitivity for the first few measurement cycles.

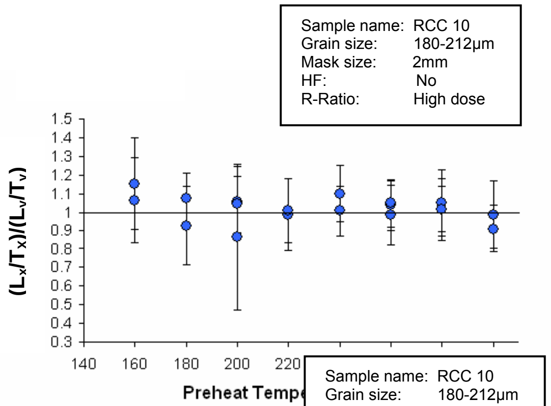
5.2.5 Recycling Ratio (R-Ratio) test

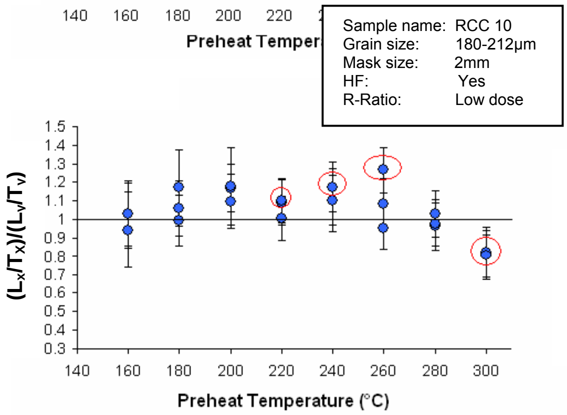
The values in figure 5.4 increase and decrease by between 25% and 35% typical of a SAR measurement cycle (Jacobs *et al.,* 2003a). For the above samples it can be assumed that sensitivity changes are corrected successfully. To check the validity of this assumption the SAR protocol makes use of the Recycling Ratio test (R-Ratio) (Murray & Wintle, 2000). The R-ratio is similar to the IR OSL depletion ratio test and uses a $[(L_2/T_2)/(L_1/T_1)]$ ratio to determine the degree of change in sensitivity. Two R-Ratio tests were performed on all the samples using the ratio $[(L_2/T_2)/(L_1/T_1)]$ to determine the rejection criteria. The first R-Ratio tests were performed using a low dose generally first administered at the beginning of the SAR measurement cycle, where one would expect to find the most sensitivity changes taking place. The second set of R-Ratios is taken from recycled points which were given a high dose. The results of these experiments are presented in figure 5.5 below. Note that the dataset does not include aliquots that were rejected using the IR-OSL depletion ration test. Most of the aliquots pass this test indicating good recycling behaviour.

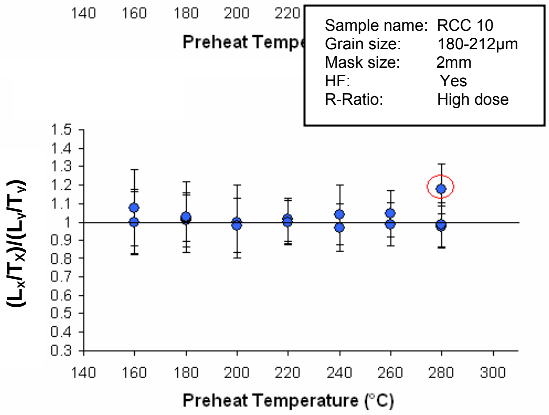

Sample name: RCC 21
Grain size: 180-212µm
Mask size: 2mm
HF: Yes
R-Ratio: Low dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

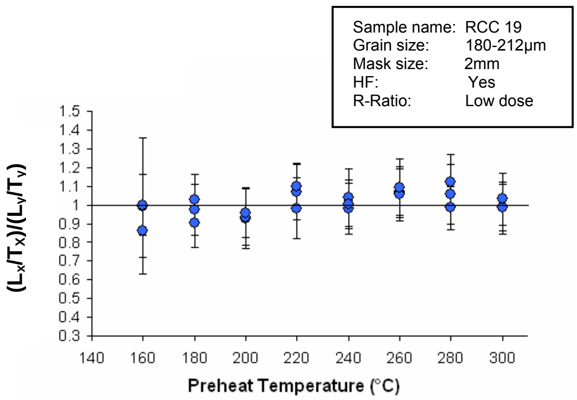

Sample name: RCC 21
Grain size: 180-212µm
Mask size: 2mm
HF: Yes
R-Ratio: High dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

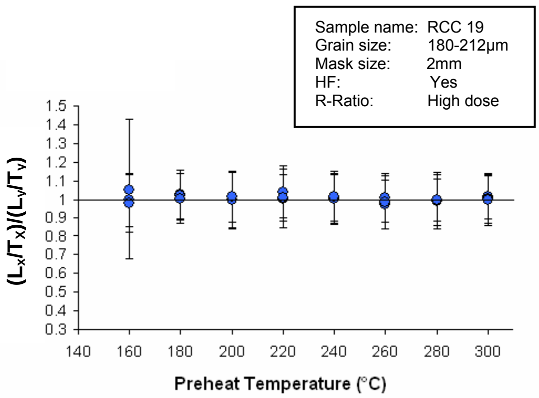

Sample name: RCC 10
Grain size: 180-212µm
Mask size: 2mm
HF: No
R-Ratio: Low dose
$(L_x/T_x)/(L_y/T_y)$

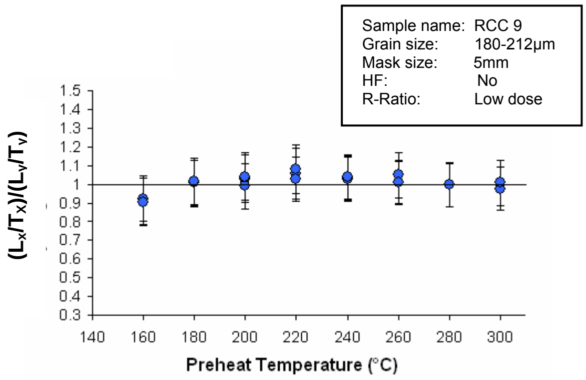

Sample name: RCC 10
Grain size: 180-212µm
Mask size: 2mm
HF: No
R-Ratio: High dose
$(L_x/T_x)/(L_y/T_y)$

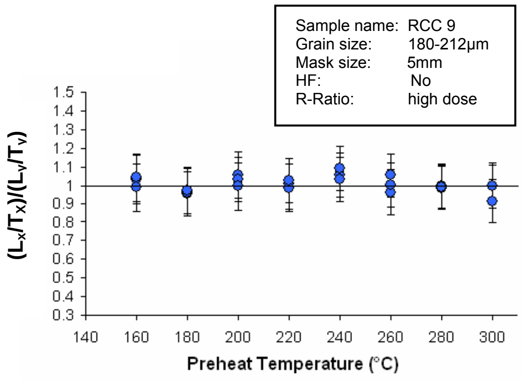

Sample name: RCC 10
Grain size: 180-212µm
Mask size: 2mm
HF: Yes
R-Ratio: Low dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

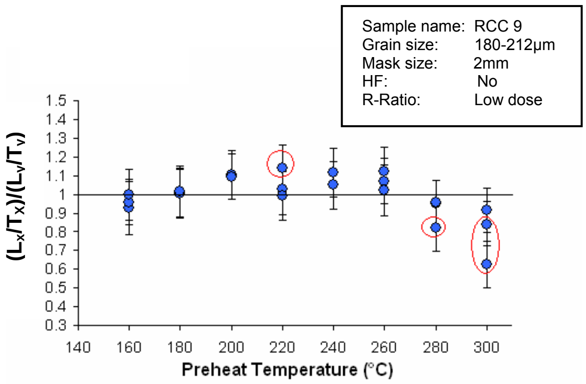

Sample name: RCC 10
Grain size: 180-212µm
Mask size: 2mm
HF: Yes
R-Ratio: High dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

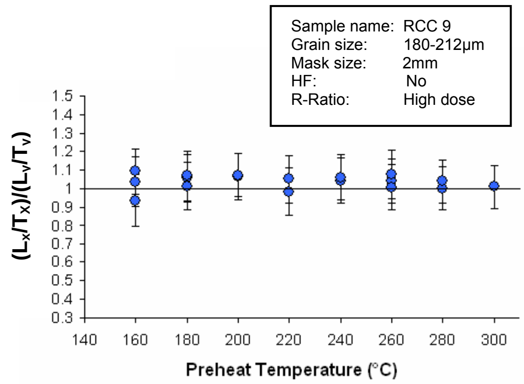

Sample name: RCC 19
Grain size: 180-212µm
Mask size: 2mm
HF: Yes
R-Ratio: Low dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

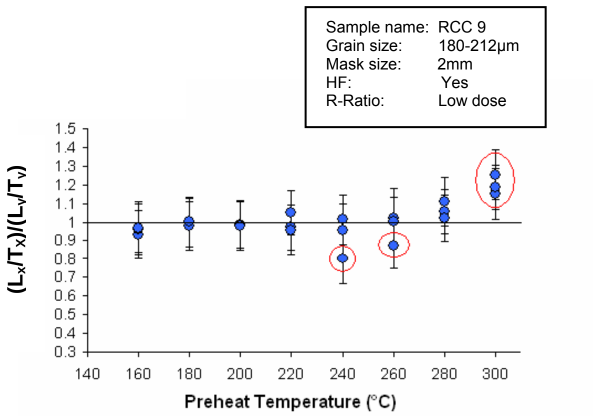

Sample name: RCC 19
Grain size: 180-212µm
Mask size: 2mm
HF: Yes
R-Ratio: High dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

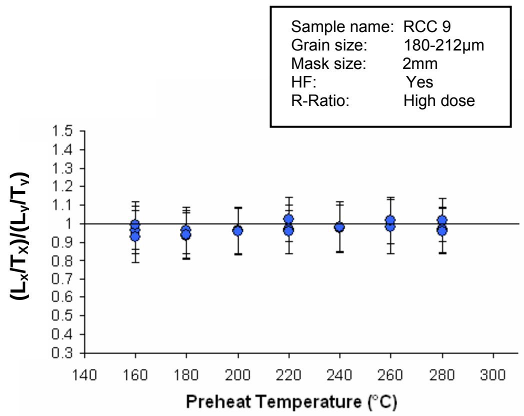

Sample name: RCC 9
Grain size: 180-212µm
Mask size: 5mm
HF: No
R-Ratio: Low dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

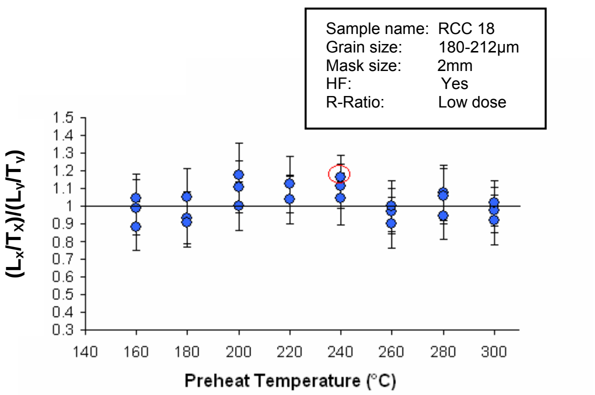

Sample name: RCC 9
Grain size: 180-212µm
Mask size: 5mm
HF: No
R-Ratio: high dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

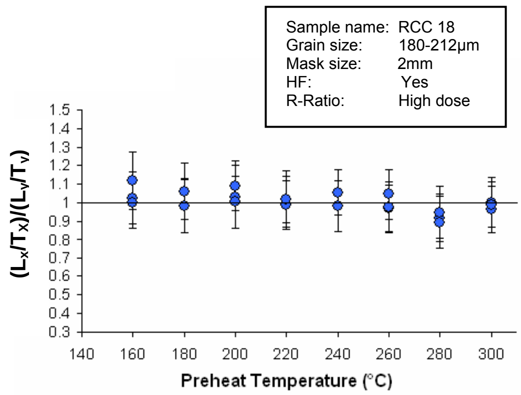

Sample name: RCC 9
Grain size: 180-212µm
Mask size: 2mm
HF: No
R-Ratio: Low dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

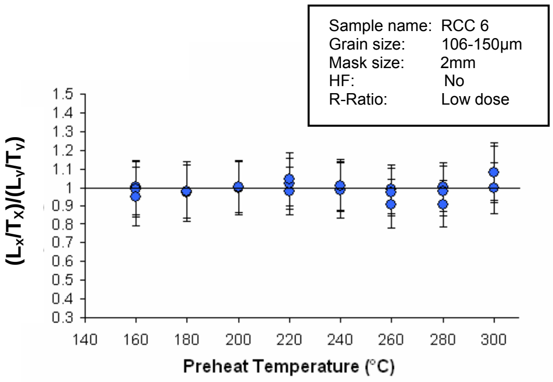

Sample name: RCC 9
Grain size: 180-212µm
Mask size: 2mm
HF: No
R-Ratio: High dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

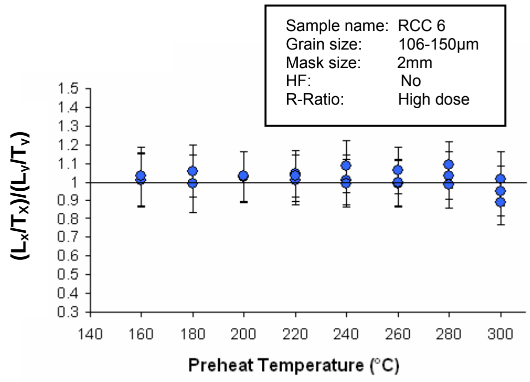

Sample name: RCC 9
Grain size: 180-212µm
Mask size: 2mm
HF: Yes
R-Ratio: Low dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

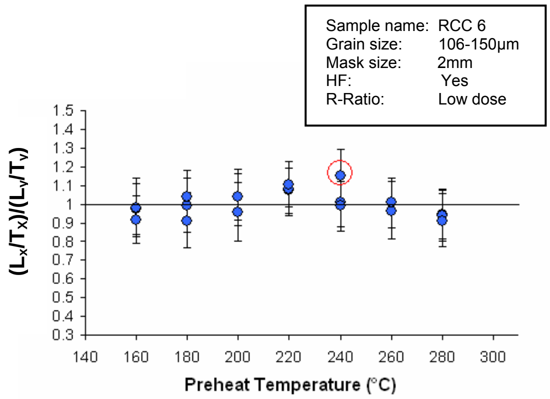

Sample name: RCC 9
Grain size: 180-212µm
Mask size: 2mm
HF: Yes
R-Ratio: High dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

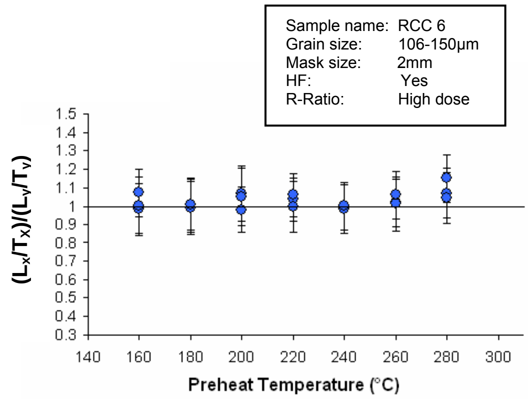

Sample name: RCC 18
Grain size: 180-212µm
Mask size: 2mm
HF: Yes
R-Ratio: Low dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

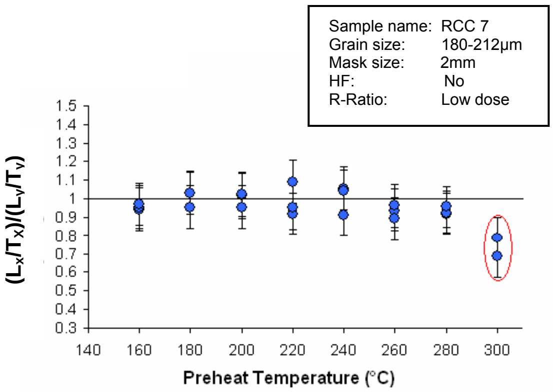

Sample name: RCC 18
Grain size: 180-212µm
Mask size: 2mm
HF: Yes
R-Ratio: High dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

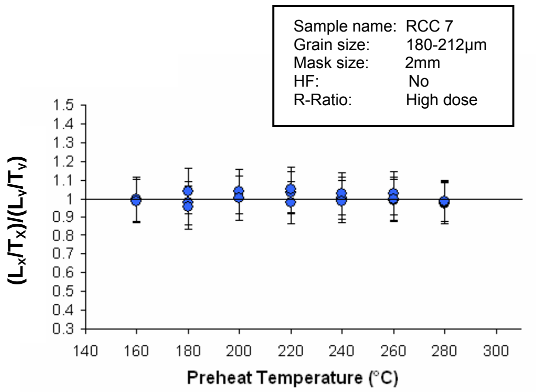

Sample name: RCC 6
Grain size: 106-150µm
Mask size: 2mm
HF: No
R-Ratio: Low dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

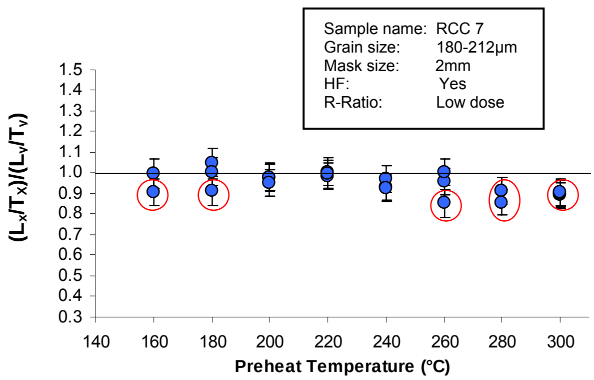

Sample name: RCC 6
Grain size: 106-150µm
Mask size: 2mm
HF: No
R-Ratio: High dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)


Sample name: RCC 6
Grain size: 106-150µm
Mask size: 2mm
HF: Yes
R-Ratio: Low dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)


Sample name: RCC 6
Grain size: 106-150µm
Mask size: 2mm
HF: Yes
R-Ratio: High dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)


Sample name: RCC 7
Grain size: 180-212µm
Mask size: 2mm
HF: No
R-Ratio: Low dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)


Sample name: RCC 7
Grain size: 180-212µm
Mask size: 2mm
HF: No
R-Ratio: High dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)


Sample name: RCC 7
Grain size: 180-212µm
Mask size: 2mm
HF: Yes
R-Ratio: Low dose
$(L_x/T_x)/(L_y/T_y)$
Preheat Temperature (°C)

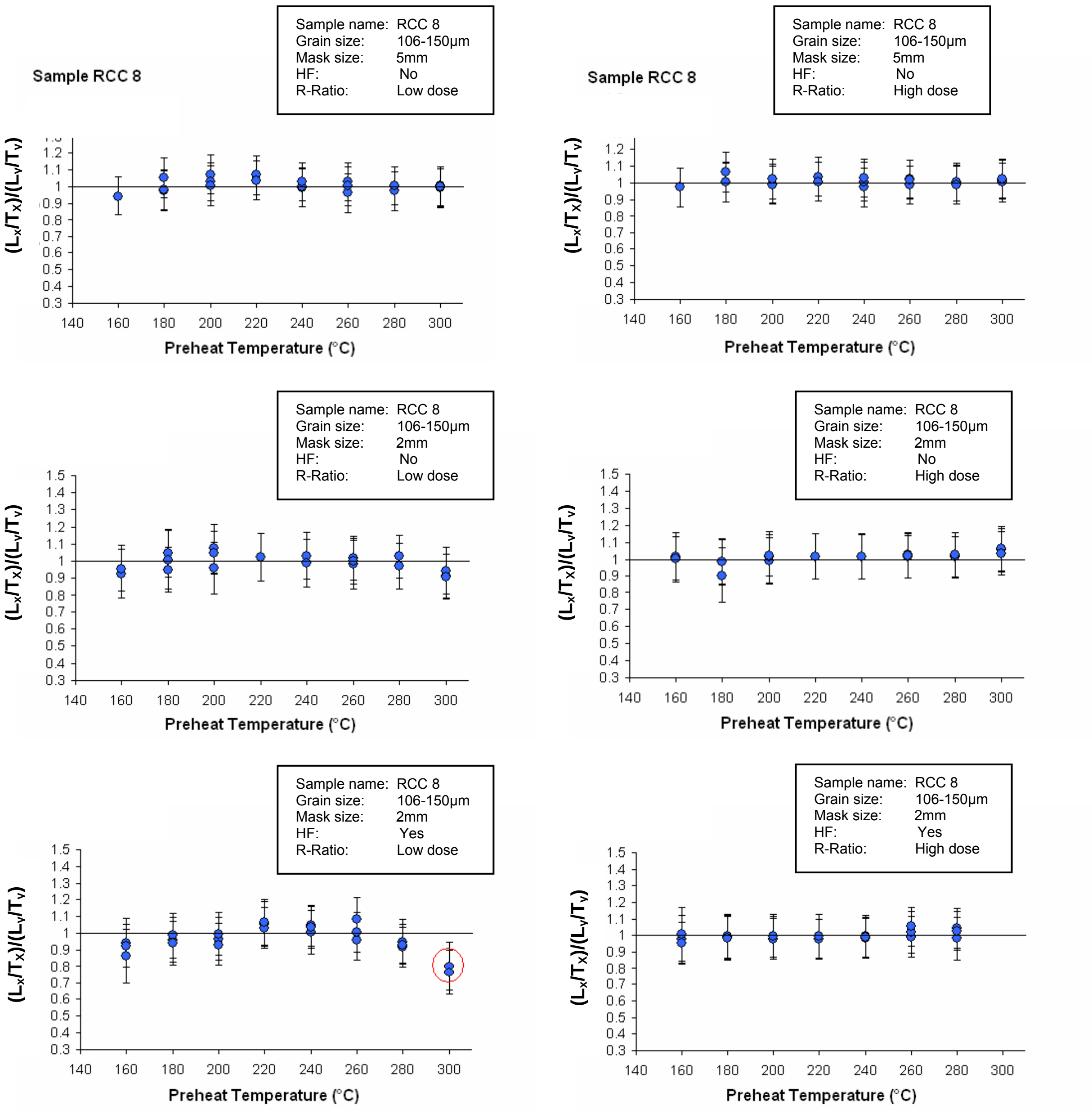


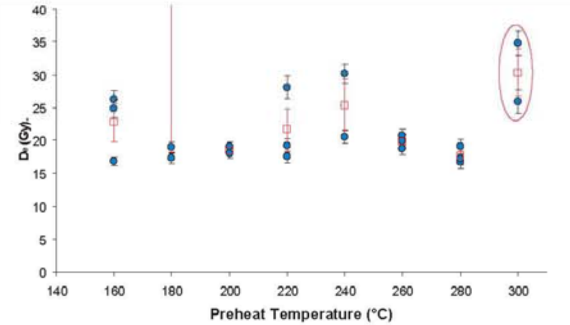
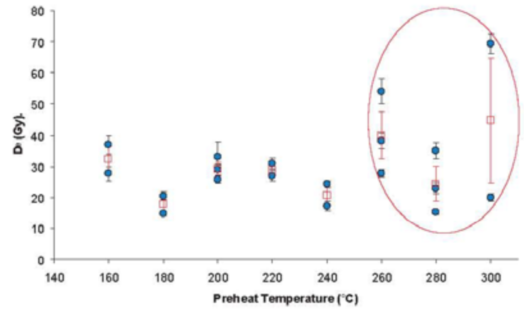
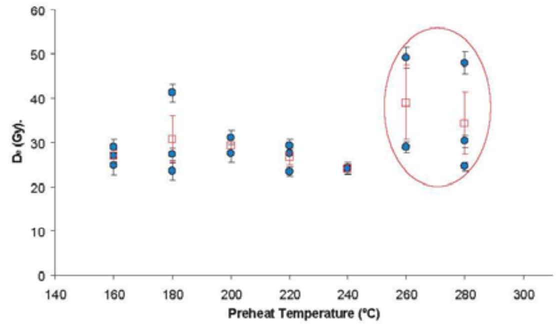
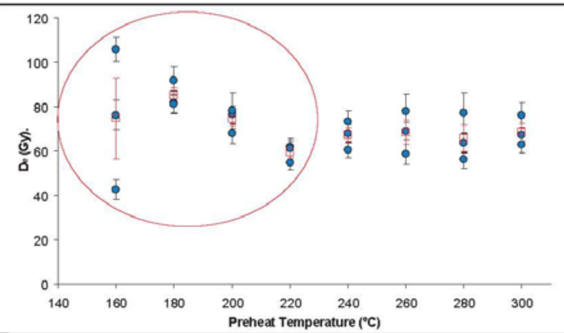
Figure 5. 5 Low and high dose recycling ratio (R-Ratio) tests performed on aliquots using different mask sizes. 24 aliquots were divided into eight groups of three and measured at different preheat temperatures. The ratio is given as $[(L_2/T_2)/(L_1/T_1)]$. No aliquots which were rejected by the IR-OSL depletion ratio test are shown.

5.2.6 Recuperation

The effects of recuperation or ‘charge transfer’ could result in unacceptable variability in D_e values (Aitken & Smith, 1988) i.e. re-trapping of the charge that has remained in traps from the previous measurement and is induced by heat treatment applied to the sample during a standard laboratory measurement protocol. Recuperation is likely to be minimal when using the conventional SAR protocol as all measurement cycles are carried out at 125°C (Murray & Wintle, 2000). Unwanted charge transfer is generally produced by traps that are emptied at higher preheat temperatures. The reliability of the corrected SAR growth curve is therefore reliant on minimal charge transfer between regenerative points (Murray & Wintle, 2000). The addition of a zero dose regenerative point visually demonstrates whether the curve is fitted through zero. A test for recuperation is used in this study by plotting the ratio between the zero dose and natural measurement and plotting the ratio as a percentage of the natural ($\{L_5/T_5\}/\{L_1/T_1\}$), all values that are greater than 5% (Murray & Olley, 2002) are rejected. No samples from RCC had recuperation values greater than 1.5%.

5.2.7 D_e versus T – Preheat Plateau

A plateau of D_e versus preheat temperature has been used in this study to ensure that reliable D_e values have been obtained for all the RCC samples. Isothermal decay curves obtained from Australian quartz using preheat ranges between 160°C and 280°C have demonstrated that the initial natural OSL signal is derived from a single trap (Wintle & Murray, 1998). The ability of the SAR protocol (Murray & Wintle, 2000) to correct for sensitivity changes is demonstrated by using a preheat range between 160°C and 300°C, if the same D_e values are obtained for all the accepted aliquots for which sensitivity changes have been corrected. In figure 5.6 the results for preheat plateaus are plotted; in most of the samples the lower and higher preheat ranges have been rejected. In a well behaved sample the charge transfer is evident at lower preheat temperatures and charge depletion occurs at higher preheat temperatures. Note that all samples in figure 5.6 do not include aliquots that have been rejected by previous rejection criteria.

<table>
<tr><th></th><th>5mm Mask size
(un-etched material)</th><th>2mm Mask size
(un-etched material)</th><th>2mm Mask size
(etched material)</th></tr>
<tr><td>RCC Sample 21
180-212 µm</td><td></td><td></td><td>
</td></tr>
<tr><td>RCC Sample 10
180-212 µm</td><td></td><td>
</td><td>
</td></tr>
<tr><td>RCC Sample 19
180-212 µm</td><td>
</td><td></td><td></td></tr>
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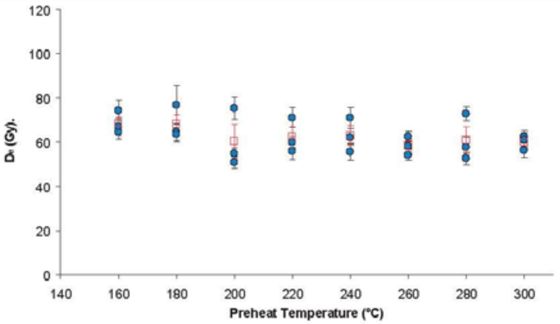
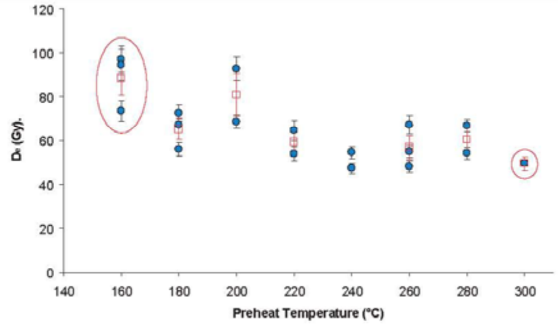
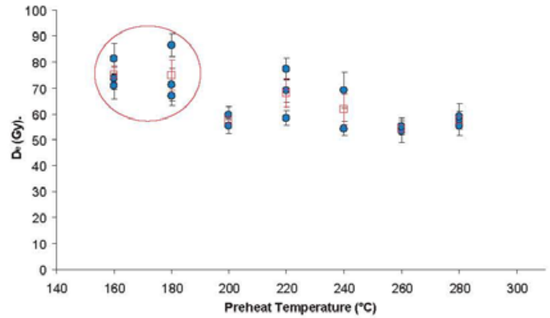
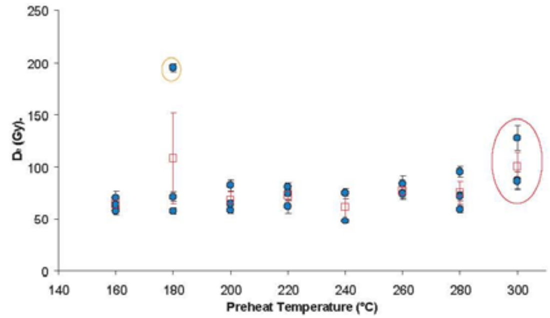
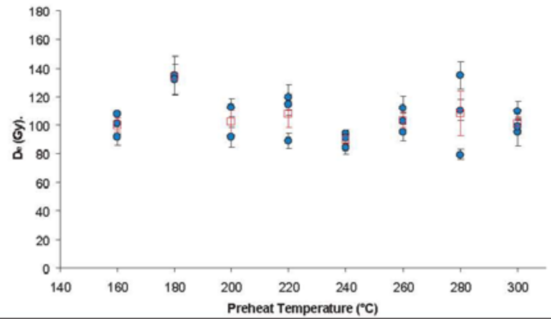
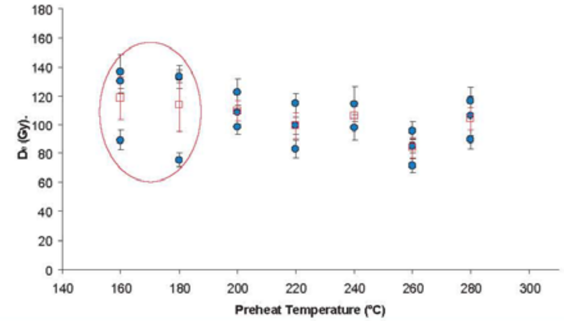
	5mm Mask size (un-etched material)	2mm Mask size (un-etched material)	2mm Mask size (etched material)
RCC Sample 9 180-212 µm			
RCC Sample 18 180-212 µm			
RCC Sample 6 106-150 µm			

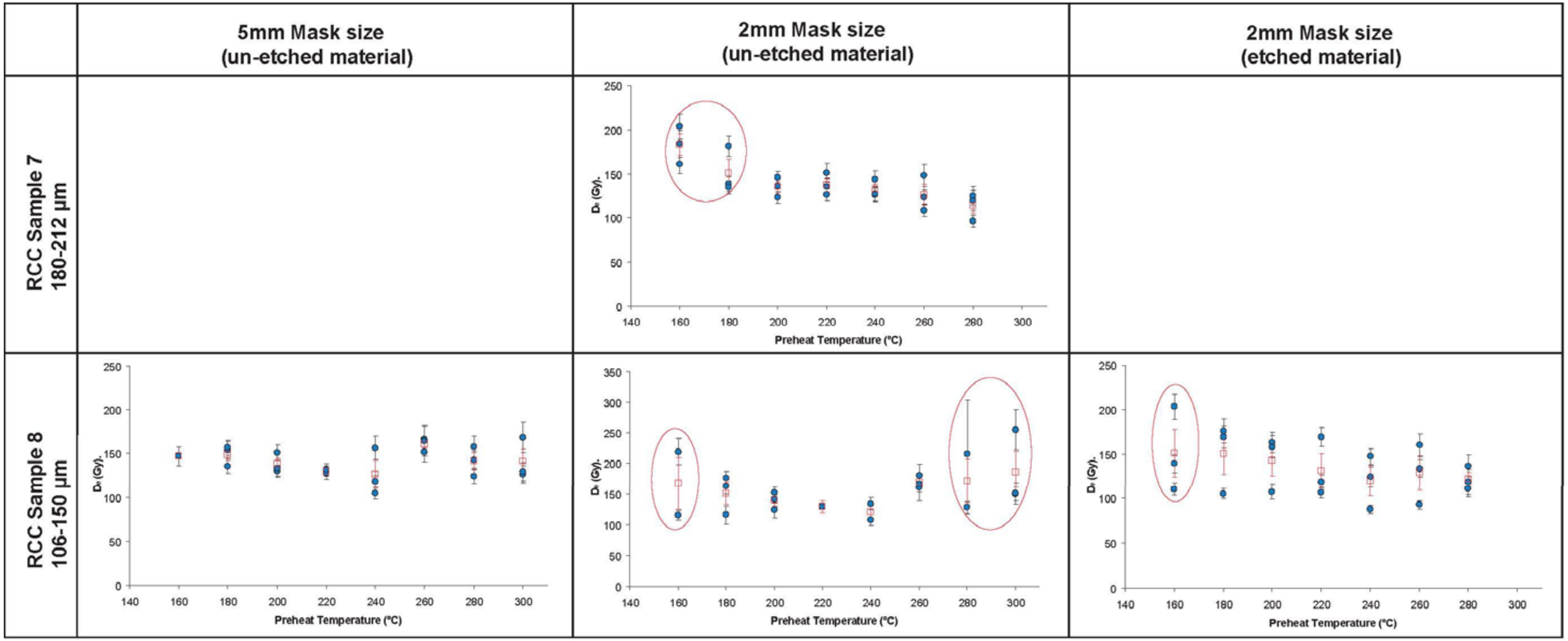
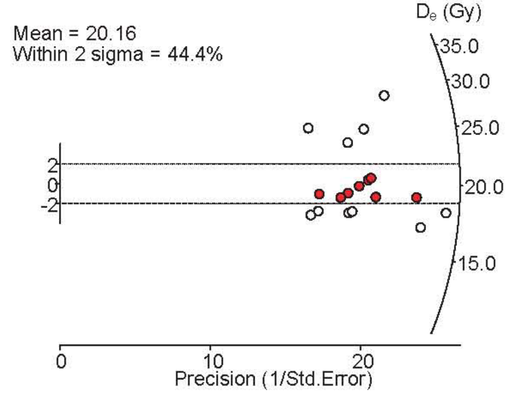
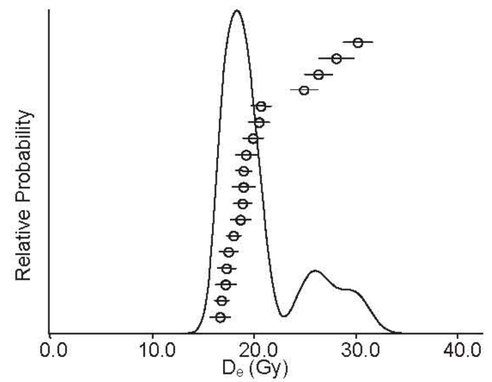
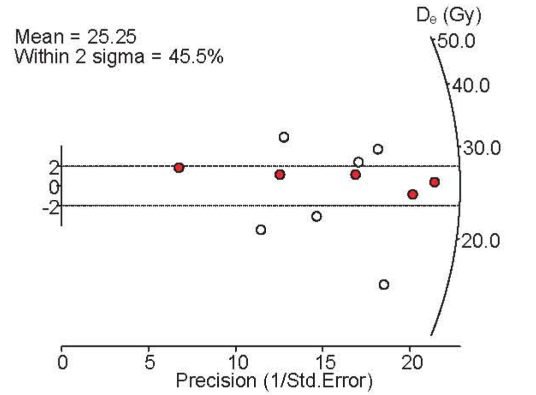
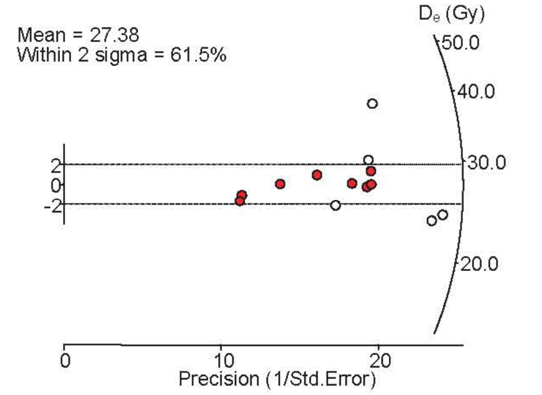


Figure 5. 6 Preheat Plateau results for RCC samples. 24 aliquots were divided into eight groups of three and measured at different preheat temperatures. The plot shows the relationship between D_e *vs Temperature.* No aliquots that were rejected by previous rejection criteria are shown.

5.3 Analysis of D_e values

Figure 5.7 and table 5.3 present the results for D_e values after all SAR criteria have been met. These values are plotted as radial plots and probability density plots. Some of D_e measurements reflect large overdispersion values that need to be explained. A potential answer to such large overdispersion could be attributed to either sample mixing or to the large percentage of feldspar grains (not detected using the IR-OSL test) present in the samples (up to 60% composition in certain layers). The IR-OSL depletion ratio tests that were applied to samples RCC 6-10 using un-etched material measured on a large 5mm mask size resulted in some samples that were completely rejected either due to feldspar contamination or bad recycling behaviour. It was therefore assumed that once feldspar contaminated aliquots were rejected the remaining aliquots would be acceptable after all the other SAR tests were performed. On all the samples a central age model is suggested due to the general scatter in distribution.

	5mm Mask size (un-etched material)	2mm Mask size (un-etched material)	2mm Mask size (etched material)
RCC Sample 21 Radial plots			
RCC Sample 21 PD plots			

<table>
<tr><th></th><th>5mm Mask size
(un-etched material)</th><th>2mm Mask size
(un-etched material)</th><th>2mm Mask size
(etched material)</th></tr>
<tr><td>RCC Sample 10
Radial plots</td><td></td><td>
</td><td>
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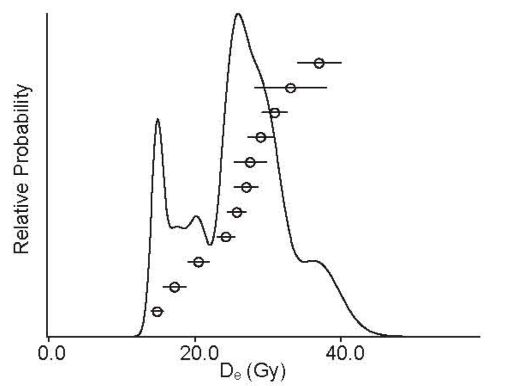
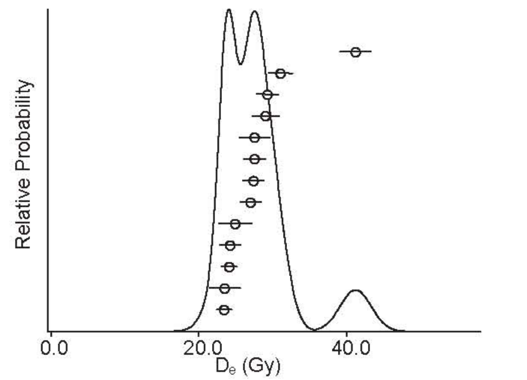
<tr><td>RCC Sample 10
PD plots</td><td></td><td>
</td><td>
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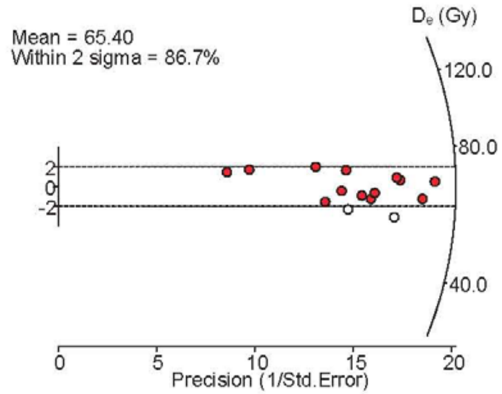
<tr><th></th><th>5mm Mask size (un-etched material)</th><th>2mm Mask size (un-etched material)</th><th>2mm Mask size (etched material)</th></tr>
<tr><td>RCC Sample 19 Radial plots</td><td></td><td></td><td>
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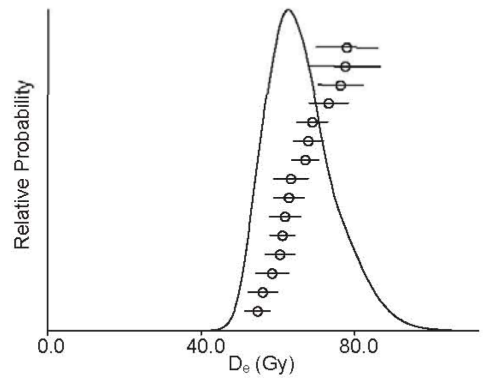
<tr><td>RCC Sample 19 PD plots</td><td></td><td></td><td>
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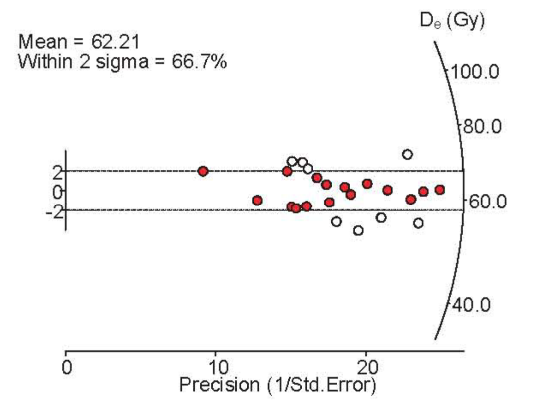
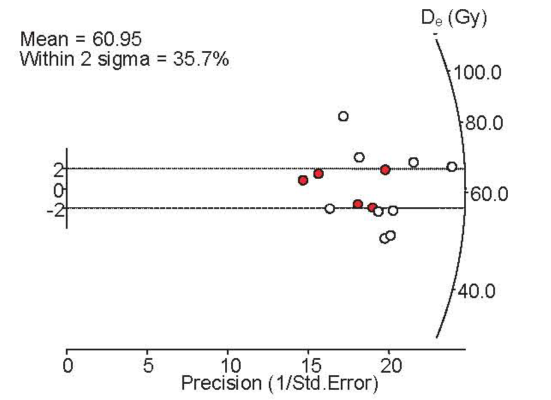
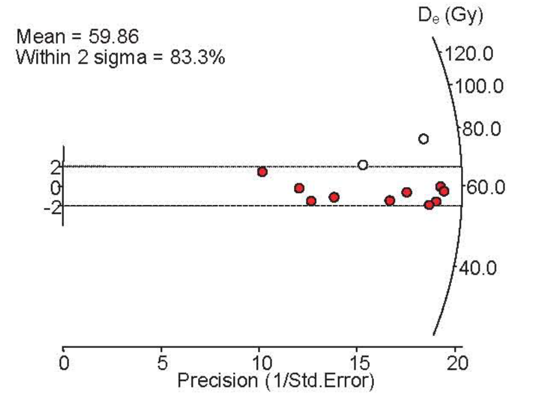
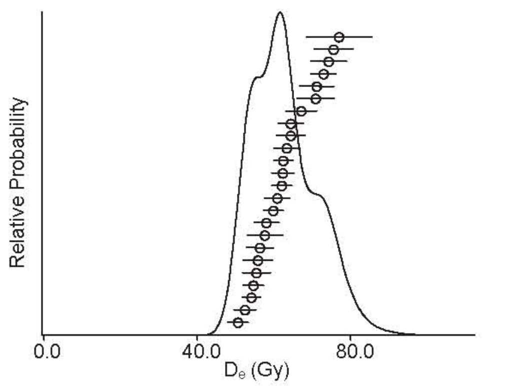
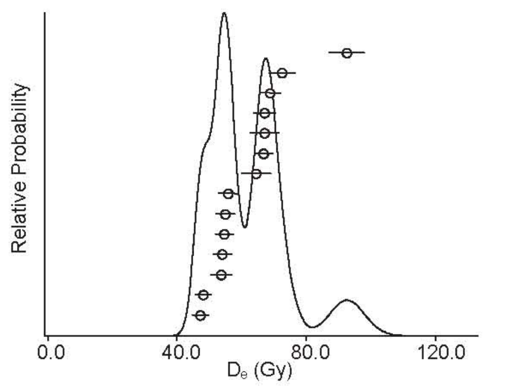
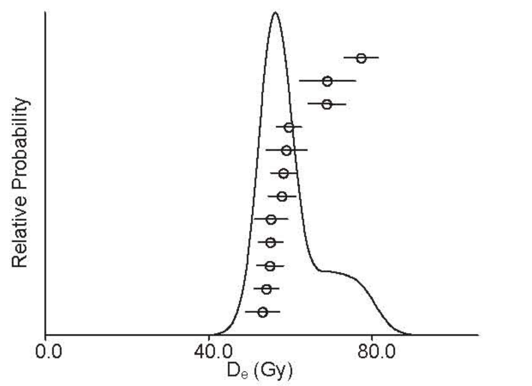
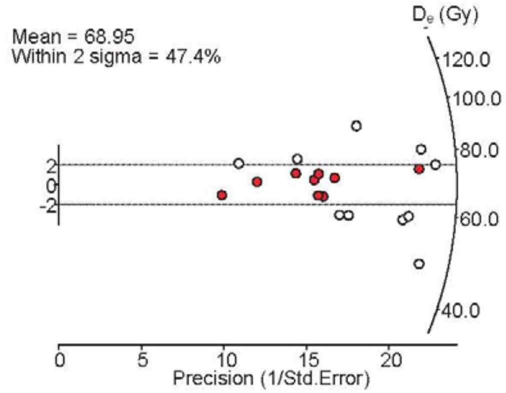
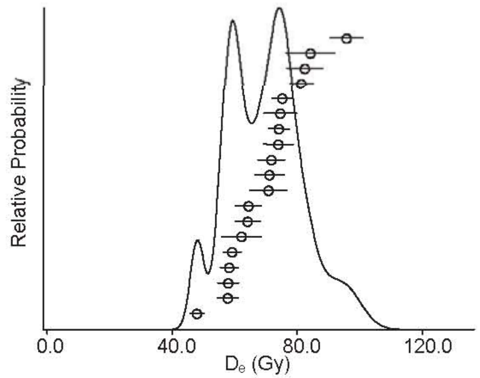
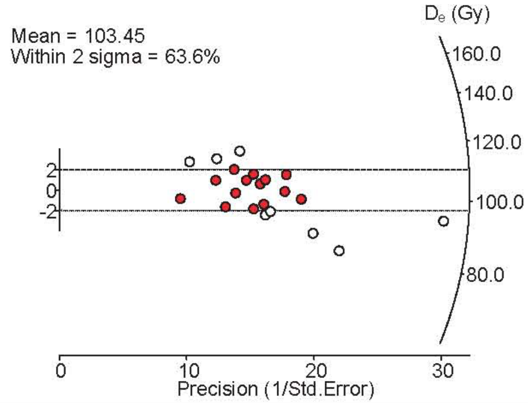
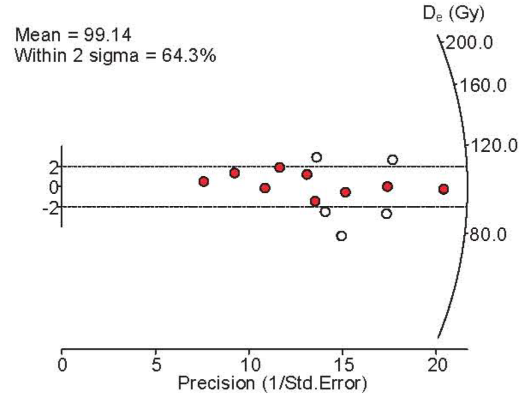
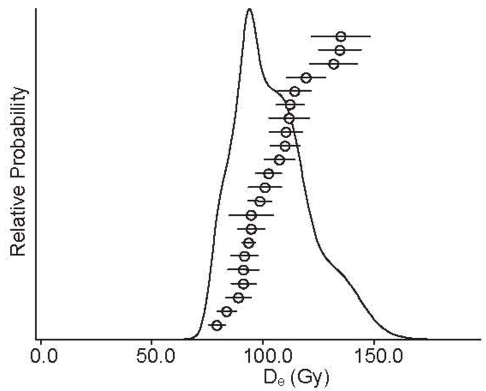
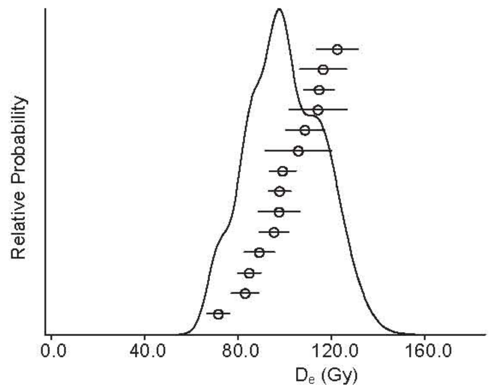
	5mm Mask size (un-etched material)	2mm Mask size (un-etched material)	2mm Mask size (etched material)
RCC Sample 9 Radial plots			
RCC Sample 9 PD plots			

	5mm Mask size (un-etched material)	2mm Mask size (un-etched material)	2mm Mask size (etched material)
RCC Sample 18 Radial plots			
RCC Sample 18 PD plots			

	5mm Mask size (un-etched material)	2mm Mask size (un-etched material)	2mm Mask size (etched material)
RCC Sample 6 Radial plots			
RCC Sample 6 PD plots			

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(un-etched material)</th><th>2mm Mask size
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(etched material)</th></tr>
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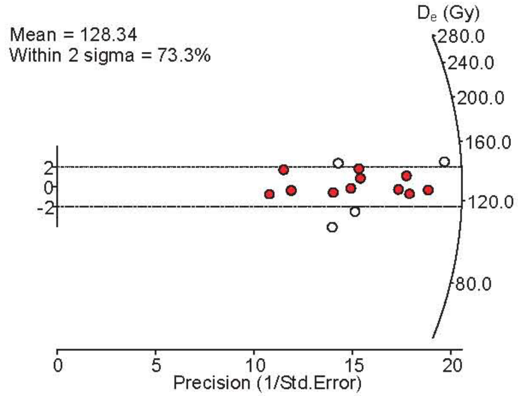
Radial plots</td><td></td><td>
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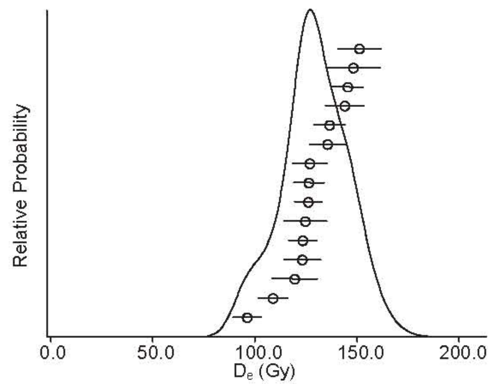
PD plots</td><td></td><td>
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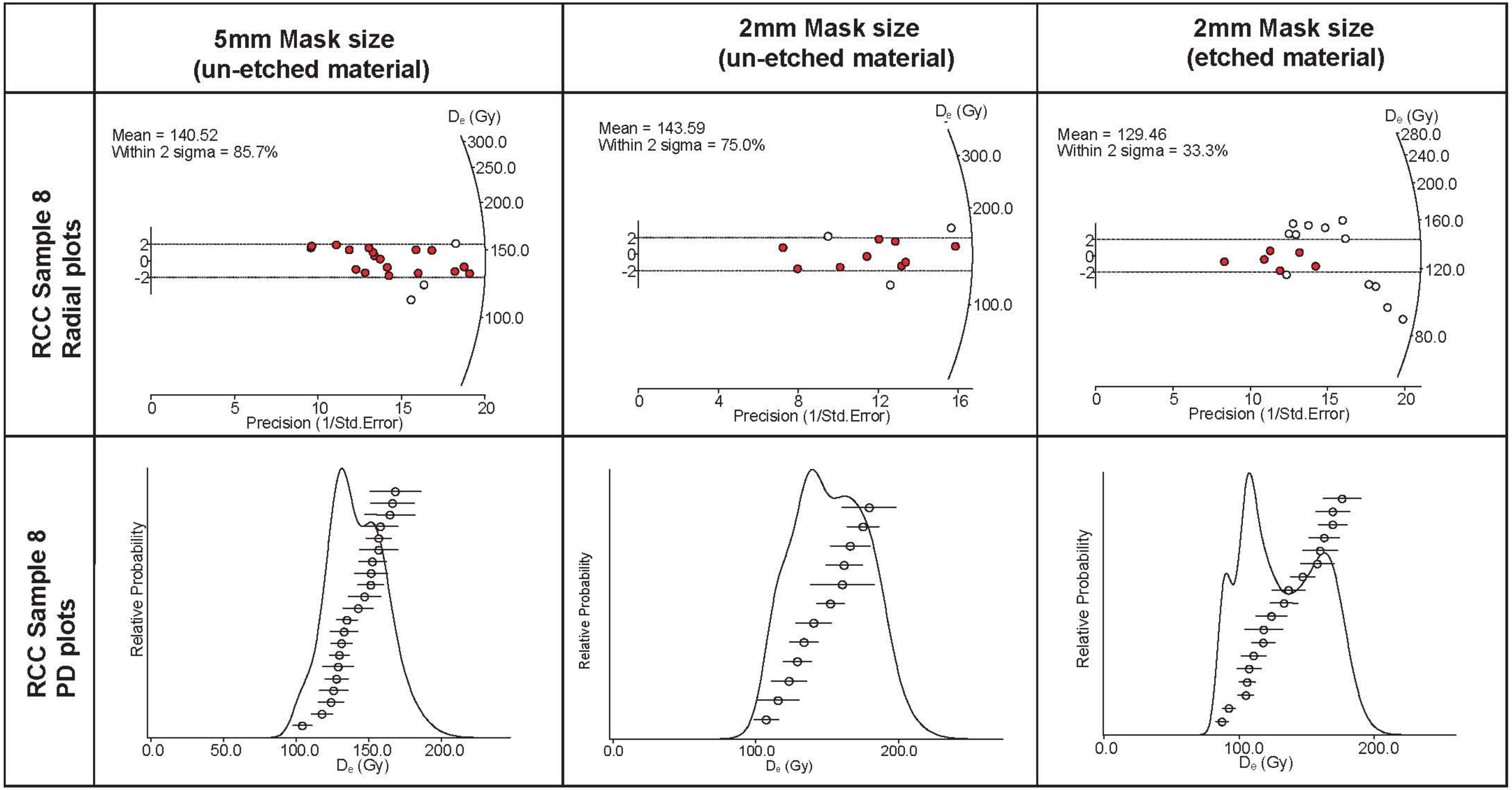

Figure 5. 7 Radial plots and PD plots showing the D_e distributions for the RCC samples. A central age model is suggested for all samples based on the general scatter in distribution.

Table 5. 3 Summary of D_e values obtained from RCC samples, measured in Pretoria.

Sample name	Sample affinities	D_e (Gy) values	Overdispersion	Comment
RCC 21	2mm mask size Etched	20.2 ± 0.8	15.8%	Mixed population or internal feldspar contamination
RCC 10	2mm mask size Un-etched	25.1 ± 2.0	25.2%	Mixed population or internal feldspar contamination
RCC 10	2mm mask size etched	27.5 ± 1.1	13.8%	Decreased scatter possibly due to feldspar elimination
RCC 19	2mm mask size etched	64.6 ± 1.6	7.2%	Good population distribution; however, does not correlate to known ^{14}C age
RCC 9	5mm mask size Un-etched	61.9 ± 1.4	9.8%	Good population distribution
RCC 9	2mm mask size Un-etched	61.0 ± 2.8	15.6%	Mixed population or internal feldspar contamination
RCC 9	2mm mask size etched	59.7 ± 2.0	9.4%	Decreased scatter possibly due to feldspar elimination
RCC 18	2mm mask size etched	68.8 ± 2.6	15.3%	Mixed population or internal feldspar contamination
RCC 6	2mm mask size Un-etched	102.7 ± 3.1	12.7%	Good population distribution
RCC 6	2mm mask size etched	98.5 ± 4.0	12.9%	Good population distribution
RCC 7	2mm mask size Un-etched	128.6 ± 3.8	9.1%	Good population distribution
RCC 8	5mm mask size Un-etched	139.3 ± 3.7	9.8%	Good population distribution; however, there is a large D_e range
RCC 8	2mm mask size Un-etched	143.9 ± 5.7	13.4%	Good population distribution; however, there is a large D_e range
RCC 8	2mm mask size etched	129.2 ± 5.7	20.7%	Mixed population or internal feldspar contamination