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Abstract

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Keywords

feldspar, infrared, stability, thermal, k, states, tail, band, luminescence, effect, stimulated, CAS

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The effect of band-tail states on the thermal stability of the infrared stimulated luminescence from K-feldspar

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Abstract

The thermal stability of the infrared stimulated luminescence (IRSL) signal from a sedimentary K-feldspar was investigated using isothermal decay study. It is observed that the isothermal decay of IRSL signal cannot be described using a first-order exponential decay function. Instead, the decay can be well described by considering the presence of band-tail states. Based on the isothermal decay results, a trap depth of ~ 1.92 eV was obtained for the IRSL stimulated at 50°C and the width of the band-tail states was found to be ~ 0.37 eV below the conduction band edge. Deeper trap depths (up to ~ 2.06 eV) were obtained for multi-elevated-temperature post-IR IRSL (MET-pIRIR) at elevated temperatures. Based on these observations, the implication for the IRSL dating feldspar was discussed.

Keywords: feldspar, luminescence, thermal stability, isothermal decay, IRSL.

1. Introduction

For optical dating utilizing feldspar grains from sediments, infrared (IR) stimulated luminescence (IRSL) signal has been commonly used (Hütt et al., 1988). However, the source of the IRSL traps and the kinetics process of IRSL is still poorly understood and controversial (e.g. Murray et al., 2009; Jain and Ankjaergaard, 2011; Li and Li, 2011). One useful way to study the source of IRSL signal is to measure its thermal stabilities using either the so-called pulse annealing (e.g. Li and Tso, 1997) or isothermal decay (e.g. Murray et al., 2009) procedures. Previous studies focused on the initial part of the IRSL signal obtained by stimulating at low temperature ($\sim 50^{\circ}\text{C}$) and suggested that the IRSL traps are associated with deep traps with a thermal depth of ~ 1.6 eV (Murray et al., 2009; Li and Tso, 1997). Recently, different luminescence behaviours for the IRSL signal as a function of IR stimulation time has been observed (Thomsen et al., 2008; Li, 2010), suggesting a change in recombination route as a function of IR stimulation time. Particularly, it was observed that the initial part of the IRSL signal has a higher anomalous fading rate when compared to the later part (Thomsen et al., 2008). This phenomenon has been explained as that the IRSL signal is a result of tunnelling recombination from the IR-excited state (Poolton et al., 2002a, 2002b). In a recent study, Li (2010) observed a strong dependence of thermal assistance energy on IR stimulation time; this can be explained as that the initial part of IRSL signal is dominated by tunnelling recombination while the later part is dominated by thermally assisted recombination via band-tail states (Poolton et al., 2002b) or conduction band. The presence of the band-tail states has been recently confirmed by Poolton et al. (2009) and it was found that the band-tail states in feldspars exist further below the high-mobility conduction band up to a band-width of ~ 0.4 eV.

More recently, the thermal stability of IRSL and post-IR IRSL (pIRIR) signal was investigated in several studies (Li and Li, 2011; Thomsen et al., 2011). Using potassium-rich feldspar (KF) grains extracted from sediments from China, Li and Li (2011) studied the thermal stability of the IRSL and pIRIR signals using a procedure of multi-elevated-temperature post-IR IRSL stimulations (MET-pIRIR), in which the IRSL signals were stimulated repeatedly at different temperatures (50-200°C). Based on their pulse annealing studies, Li and Li (2011) suggested that the MET-pIRIR signals at elevated temperatures (>100 °C) are more thermally stable than the IRSL signal at 50°C, and this was explained as the presence of deeper traps in the MET-pIRIR signals. Similar results were also reported by Thomsen et al. (2011) who showed that the pIRIR signal has a greater thermal stability than the IRSL. They concluded that their data can be explained in terms of either a single-trap with the presence of band-tail states or the presence of multi-trapping levels. Using time-resolved stimulation study, Jain and Ankjærgaard (2011) showed that the IRSL signal at low temperature and the post-IR IRSL (pIRIR) signal at elevated temperatures have a different recombination routes via band-tail states; the former are dominated by recombination of spatially close electron-hole pairs and the latter are dominated by recombination of distant electron-hole pairs. Based on their observations, they argued that the observed difference in the thermal stabilities of IRSL and pIRIR signals can be explained as a single-trap model with the presence of band-tail states.

It has been suggested that thermal hopping among the band-tail states plays an important role in the production of IRSL from feldspar (Poolton et al., 2002a, 2002b, 2009). As a result, the electrons can move with a limited mobility and recombine with holes. A significance of the presence of band-tail states is that the stability of trapped charges would be considerably lower than that expected if the band-tail states did not exist. This implies that the

trap depth of IRSL signal might have been underestimated in previous studies (Murray et al., 2009; Li and Tso, 1997; Li and Li, 2011). Hence, it is important to re-consider the presence of band-tail states in the study of thermal stability and the sources of IRSL traps.

In this study, we further study the thermal stabilities of IRSL and pIRIR signals from KF. We showed that the isothermal decay of the IRSL and pIRIR signals cannot be described using a first-order exponential decay function of a single trap, but it can be well described by assuming the presence of band-tail states.

2. Samples and experimental details

An eolian sedimentary sample (Sm1) from the transition zone between the Mu Us Desert and the Loess plateau in central China were used in this study. This sample has been extensively studied previously (Li and Li, 2008, 2011) and has typical luminescence features commonly observed from sedimentary K-feldspars in different regions of North China. The samples were routinely treated with HCl and H₂O₂ to remove carbonate and organic matter in subdued red safe-light conditions. After drying, 150-180 μm grains were obtained by sieving. The KF grains were separated using heavy liquids. The extracted KF grains were cleaned using 10% HF for 40 minutes. Aliquots containing several hundred grains were prepared by mounting the grains in a monolayer on a 9.8 mm diameter aluminum disc with “Silkospay” silicone oil. The IRSL measurements were made on an automated Risø TL-DA-12 reader equipped with IR diodes (880 nm) for stimulation. Irradiations were carried out within the reader using a ⁹⁰Sr/⁹⁰Y beta source. The IRSL signals were detected through a filter pack containing Schott BG-39 and Corning 7-59 filters, which allows for a blue violet transmission (320-480 nm).

3. Results

3.1 Isothermal decay

3.1.1 Theoretical consideration

Here we describe the isothermal decay of IRSL signals by considering the presence of the band-tail states. Several assumptions were made in this model:

- 1) Following Poolton et al. (2002b, 2009), it is assumed that the density (ρ) of band-tail states (E_b) is exponentially distributed below the conduction band edge (E_C), i.e.

$$\rho(E_b) = \rho_0 \exp\left(-\frac{E_b}{E_u}\right)$$

where ρ_0 is the density of the sub-conduction band states ($E_b \approx E_C=0$) and E_u is the Urbach band-tail width. It is noted that other distributions such as linear, Gaussian and double-exponential could be possible (Chen and McKeever, 1997).

- 2) The probability of electrons thermally evicted into the band-tail states of energy in the range of (E_b+dE_b) , $P(E_b)dE_b$, is proportional to the density of the band-tail states $\rho(E_b)$, then it follows

$$P(E_b)dE_b = A \exp\left(-\frac{E_b}{E_u}\right) dE_b \quad (1)$$

where A is a constant. It is assumed that that all recombination during thermal stimulation occur only through the band tail states, and the excited state of the dosimetric trap or the conduction band do not play any role in charge recombination.

- 3) A first-order kinetic condition is assumed, i.e. the cross-section of retrapping is negligible after the trapped electrons being thermally released into the band-tail states or

conduction band before they subsequently recombine with holes, either via thermal hopping or via tunneling.

Based on the above assumptions, for the IRSL traps with an energy depth of E_t , the lifetime (τ) of thermal eviction of the trapped electrons into band-tail states (with energy of E_b) during isothermal measurement can be written as:

$$\tau = s^{-1} e^{\left(\frac{E_t - E_b}{kT}\right)} \quad (2)$$

where s is the frequency factor, k is the Boltzmann constant, T is the absolute isothermal temperature (K). The number of trapped electrons remaining after a time t , $n(t)$, is found by intergrating the probability of recombination via the band-tail states at $(E_b + dE_b)$ multiplied by the probability that thermal detrapping has not occurred, $\exp(-t/\tau)$. Hence, denoting n_0 to the initial trapped electron population, we have

$$\frac{n(t)}{n_0} = - \int_0^{\Delta E} A \exp\left(-\frac{E_b}{E_u}\right) \exp\left(-st * e^{-\frac{E_t - E_b}{kT}}\right) dE_b \quad (3)$$

where ΔE represents the energy of the deepest band-tail states thermally accessible, e.g. this value should be smaller than energy depth of the IRSL traps, E_t . Poolton et al [12] obtained a value of ~ 0.4 eV for E_u using their feldspars. We will show in the next section that our data yield a similar value of E_u .

3.1.2 Isothermal decay results

To test the theory in the above section, isothermal decay experiments of IRSL and MET-PIRIR was conducted. The detailed experimental procedure was listed in Table 1. One KF aliquot was first undergone five cycles of irradiation (~ 20 Gy) and TL measurement to 500°C . It was observed that such irradiation/heating cycles can stabilize the IRSL sensitivity

changes to a negligible level. The aliquot was then given a regenerative dose of 18 Gy before heating to a specific temperature (in the range of 300-360°C) and holding at this temperature for various periods. After that, five successive MET-pIRIR measurements were conducted at 50, 100, 150 200 and 250°C, respectively. A test dose of ~8 Gy was then given and the MET-pIRIR signals from test dose were measured for monitoring any sensitivity change. This cycle was repeated for a range of isothermal temperatures and periods. It is to be noted that negligible sensitivity change through measurement cycles was observed from the test dose IRSL signals.

Fig.1 shows the normalized IRSL and MET-pIRIR signals remained after isothermal heating at 300°C for different periods. It is observed that the MET-pIRIR signals measured at elevated temperatures are more thermally stable than the IRSL signal measured at 50 °C. This result is consistent with the pulse annealing result obtained by Li and Li (2011) using the same sample. The basic feature of this figure shows that none of the isothermal decay curves follows a first-order exponential decay of a single trap (note the log scale of y-axis). It is interesting to note that if we try to fit the isothermal decay curves using the first-order exponential decay, two components of exponential decay are necessary to fit the curves. However, all the trap depths obtained are very low (in the range of 1.2-1.5 eV), which is unlikely for feldspar.

Since it has been shown that the presence of band-tail states plays an important role on the kinetics behaviour of luminescence from feldspar (Poolton et al., 2009), the isothermal decay data were analysed using equation (3) described in the above section. The NAG integrator from the NAG library was used to fit the data using Origin software. It is noted that a fixed s value of $1 \times 10^{13} \text{ s}^{-1}$ was used in this study, and variation in the fitting results is expected if a significantly different s value is used, for example, using s value of $1 \times 10^{10} \text{ s}^{-1}$ in

fitting will give lower results in the energy of ground state (E_t) (~ 0.3 eV lower than the results obtained using $1 \times 10^{13} \text{ s}^{-1}$) but give similar band-tail state width (E_u). However, we adopt the value of $1 \times 10^{13} \text{ s}^{-1}$ to make our results to be comparable with previous studies (e.g. Poolton et al., 2009). This value is also close to the values experimentally determined in previous studies (e.g. Duller, 1997; Li and Tso, 1997). To further reduce the number of variables in the fitting procedure, the upper bound of the integral in equation (3), ΔE , was fixed at 1.5 eV. This value is determined to be large enough so that there is no change in the fitting results by further increasing the integral range (0-1.8 eV). This value ensures that nearly all of the charges in the IRSL traps are recombined via the band-tail states within the integral range, e.g. 0-1.5 eV. Hence, there are actually three variables in the fitting procedure, E_t , E_u and A .

The isothermal decay curves of individual IRSL and MET-pIRIR signals from 50 to 250°C from all heating temperatures (300, 320, 340 and 360°C) were shown in Fig. 2a-e, respectively. It is shown that all the data sets are well described using the equation (3) (dashed lines in the figures). The fitted values for the trap depths (E_t) and band-tail width (E_u) of band-tail states from Fig. 2 are summarized in Table 2. The fitting of the data sets for 50°C IRSL (Fig. 2a) gave similar values of parameters E_t and E_u for different isothermal temperatures. The mean value of the trap depth of 50°C IRSL given by fitting is 1.92 ± 0.02 eV. It is interesting that such a value is higher than the previous estimates (~ 1.6 eV) without consideration of band-tail states (Murray et al., 2009; Li and Tso, 1997; Li et al., 1997). Moreover, fitting of the isothermal data using equation (3) can give an estimate of the Urbach width of band-tail states (E_u), i.e. the data sets in Fig. 2a gave an estimate of 0.38 ± 0.04 eV. This value is entirely consistent with the value ~ 0.4 eV obtained by Poolton et al. (2009) from different experimental procedures, although this value might be expected to vary from sample to sample.

If we apply the same fitting procedure to all the isothermal decay results for different MET-pIRIR signals using equation (3), an interesting result was observed that the trap depth E_t increases with the stimulation temperatures of MET-pIRIR signals. The values obtained for the MET-pIRIR signals at 100, 150, 200 and 250°C are 1.95 ± 0.02 , 1.97 ± 0.02 , 2.02 ± 0.02 and 2.06 ± 0.02 eV, respectively (Fig. 2b-2e). In contrast, the values of the width of band-tail states (E_u) obtained are consistent to each other within uncertainties, and the overall mean is 0.37 ± 0.09 eV.

4. Discussions

Previous studies based on pulse annealing measurements and isothermal measurements does not measure the trap's true thermal stability (trap depth), since there was no consideration of the presence of band-tail states. It was suggested that these thermal heating studies measure the stability of electron-hole pairs, which is because a higher preheat temperature or a longer heating time will sweep out a greater volume of electron-hole pairs, and therefore, result in a lower recombination probability for the subsequent IRSL measurement (Jain and Ankjaergaard, 2011). In this study, we showed that the trap depth can be obtained by considering the presence of band-tail states, provided that the three assumptions in section 3.1.1 are met. Equation (2) predicts that the transition from the ground state into the lower part of band-tail states (with a higher value of E_b) requires a lower thermal energy. Hence, the more electrons are proximate to these low band-tail states, the lower thermal stability or lifetime they have. Since the low-energy electrons in the lower part of band-tail states have a lower mobility, a more localised recombination was expected for them. As a result, the initial part of isothermal decay curve (relative unstable electrons) represents the recombination from relatively close electron-hole pairs or localised recombination. Therefore, the difference in the thermal stability of electron-hole pairs with different distance

is mainly an apparent result of presence of band-tail states. It is therefore suggested that the trap depth estimation for K-feldspar IRSL should take such effect into account by using equation (3).

Equation (3) is obtained based on the assumption that there is no strong retrapping of charges, i.e. first-order kinetics. Actually, this assumption can be tested by investigating if there is a strong dependence of isothermal decay rate on the initial trapping population (n_0), since a strong retrapping of electrons during stimulation will result in a non-first-order kinetics, which is dependent on the initial trapping population for both delocalized recombination (Garlick and Gibson, 1948; Chen and McKeever, 1997) and localized recombination (Kumar et al., 2006). It is therefore expected that varying the regenerative dose in the isothermal study will change the isothermal decay rate significantly, which is similar to the phenomena of shifting thermoluminescence (TL) peak in a non-first-order kinetics (Chen and McKeever, 1997). To test this, we measured the isothermal decay curves using different regenerative doses varied by a factor up to 20. It was found that the experimental isothermal decay patterns did not vary significantly with different doses or initial trapped populations (data not shown), suggesting that the isothermal process was dominated by the first-order kinetics for our sample and there is a negligible retrapping of charges.

The result in Fig. 1 shows that the isothermal decay of IRSL signal cannot be described using a first-order exponential decay function of a single trap. In stead, the decay shape can be well described by first-order kinetics after considering the presence of band-tail states (Fig. 2). One of the major effects of the presence of band-tail states is that, the electrons can be thermally evicted into these states below the high-mobility conduction band. As a result, the lifetime of the trapped electrons is shorter than that to be expected without band-tail states. Our results show that the energy depth of traps related to the 50°C IRSL is ~ 1.9 eV,

which is higher than the value of ~ 1.6 eV obtained by previous studies (e.g. Murray et al., 2009; Li and Tso, 1997; Li et al., 1997). This discrepancy can be reasonably taken account by the presence of band-tail states with an energy band width of 0.3-0.4 eV obtained by fitting the isothermal decay data (Fig. 2), and this value is entirely consistent with the result obtained by Poolton et al. (2009) based on different experiments. It is noted that if the thermal depths obtained in Fig. 2 are transferred to optical depths, the IRSL trapping levels are expected to be in the range of 2.4-2.6 eV (using an optical to thermal depth ratio of 1.25 used by Hütt et al. (1988)) below the conduction band, which is consistent with the result obtained by Baril and Huntley (2003), who proposed that the IRSL trap is at least 2.5 eV below the conduction band.

The isothermal decay of MET-pIRIR signals can also be described using equation (3). The trap depths of the corresponding traps obtained are higher than the 50°C IRSL. For the 250°C MET-pIRIR signal, a value of ~ 2.1 eV in the trap depth was obtained, which is ~ 0.15 eV higher than that of 50°C IRSL. The discrepancy between the 50°C IRSL and 250°C MET-pIRIR signals could be explained by the presence of multi-trap levels. However, a single-trap model (Jain and and Ankjaergaard, 2011) still cannot be ruled out. The higher thermal stability of post-IR IRSL signals could be a result of the preferential removal of unstable charges, which are more proximate to the low-energy band-tail states (hence have short lifetime), by the prior low temperature IR stimulations. This process may lead to more trapped charges that are proximate to the higher energy part of the band-tail states (hence have longer lifetime) to be sampled by the post-IR IRSL. This discrepancy can also be a result of imperfect modelling of the isothermal process, e.g. the assumed density distribution of band-tail states and the role of thermally excitation to excited states (Mandowski, 2005) are not considered in this study. Future studies on the role of the excited state during thermal stimulation are necessary, but these rely on the knowledge of the thermal energy level of the

excited state which is poorly understood. However, the simple model proposed here can describe the thermal stability very well at different temperatures investigated, which provides a potential to incorporate the band-tail states into further studies on the kinetic process of feldspar.

5. Implications for luminescence dating

One of the critical assumptions in OSL dating is that the trapped electrons used for dating are stable over the age being dated. It is thus important to ensure that the IRSL signals are coming from deep traps that have a long kinetic lifetime at ambient temperature (e.g. $\sim 20^\circ\text{C}$). To test the effect of thermal stability to IRSL dating using feldspar, the thermal stabilities of the 50°C IRSL and 250°C MET-pIRIR signals at 20°C were simulated using equation (3) based on the parameters obtained for the trap depths ($E_t=1.92$ and 2.06 eV) and band-tail states width ($E_u=0.37$ eV) and s value of $1 \times 10^{13} \text{ s}^{-1}$. The loss of trapped charges as a function of time was shown in Fig. 3. It is shown that the trapped charges decrease quickly during the first hundreds years. The rate of losing trapped charges decrease significantly with time (note the log scale in time in Fig. 3), but a majority of the initial charges are still remaining even after one million years. This pattern is expected, because the lifetime of the charges from the ground states to the deeper parts of the band-tail states could be very short, even at ambient temperatures, which is mainly determined by the energy gap between the ground states and the band-tail states. As a result, these ‘thermally unstable’ charges deplete quickly and the charges remaining are more stable as they require higher energy (thus longer lifetime) to transfer to the band-tail states.

The pattern in Fig. 3 suggests that a part of the IRSL and MET-pIRIR signals can be thermally unstable and they are expected to be lost during the geological periods. However, to

be able to accurately date the sediments, the signal measured must be thermally stable over the geological period of interest, e.g. in the last one million years where OSL dating is usually applied. Fortunately, in luminescence dating a preheat procedure is routinely applied to remove the thermally unstable signals, i.e. heating the sample to a specific temperature for a specific time (e.g. 10 s) before the luminescence signal is to be measured. It is thus required to ensure that a preheat condition can satisfy the removal of unstable charges without eliminating the main charge population severely.

Based on the equation (3), we investigate the effect of preheat in IRSL dating of feldspar. Fig. 4 (a) and (b) show the remaining IRSL at 50 °C and MET-pIRIR 250 °C signals after preheating to different temperatures for 10 s. It is shown that preheat can result in removal of unstable charges and leave more stable charges. The higher preheat temperature is, the more unstable charges are removed. To allow for dating of a 10 ka sample with an age underestimation less than 5%, a preheat of ~200°C for 10 s is required as a minimum thermal treatment. However, a more severe preheat is required for dating older samples without considerable underestimation, e.g. a preheat at ~ 260°C for 10 s can secure dating of samples of the last one million years with an age underestimation less than 5%. It is noted that thermal stability for the MET-pIRIR 250 °C signals is not a problem for dating, because a preheat temperature as high as 300 °C is usually adopted to eliminate the interference of isothermal TL during measuring the MET-pIRIR signals at 250 °C.

Our results predict that an increase in age or D_e value should be expected in a preheat plateau test for feldspar IRSL, especially at lower preheat temperatures (<200 °C). To test this, we measured the equivalent dose using the 50°C IRSL signal with a range of preheat temperatures, from 100 to 280 °C, based on a single-aliquot regenerative-dose protocol for feldspar (Blair et al., 2005). The results are shown in Fig. 5. It clearly shows an increase in D_e

for the preheat temperatures from 100 to 200°C, which strongly support the expectation predicted from our model. A similar trend of increase in D_e with preheat temperature was also observed by Arnold et al. (2003) and Steffen et al. (2009). However, not all samples display a D_e dependence of preheating temperature. For example, for the three samples studied by Murray et al. (2009), two of them show an increase in D_e with preheat temperature up to ~200 °C, but another show a flat preheat plateau. These results suggest that the thermal stability, which is dependent on the density and energy distribution of the band-tail states, may be variable from sample to sample. A preheat plateau test should thus be conducted to find best preheat temperature for IRSL dating.

6. Conclusions

Based on our results, the following conclusions for the thermal stability and kinetic process of IRSL from K-feldspar can be obtained:

- 1) The main IRSL and the MET-pIRIR traps locate at least ~1.9 eV below the high-mobility conduction band.
- 2) The band-tail states extend further below the conduction band with a band-width of 0.3-0.4 eV.
- 3) The presence of band-tail states can cause a lower stability of trapped charges than that expected if the band-tail states did not exist.
- 4) For dating applications, appropriate preheat is necessary to equalize thermal erosion of IRSL traps in the natural and laboratory irradiated aliquots..

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Figure captions

Figure 1: The normalized IRSL and MET-pIRIR signals as a function of isothermal heating time at 300°C. All data points were normalized to the initial value.

Figure 2: Isothermal decay results from the IRSL and MET-pIRIR signals at different temperatures obtained using the procedure in Table 1. (a) IRSL at 50°C; (b) MET-pIRIR at 100°C; (c) MET-pIRIR L at 150°C; (d) MET-pIRIR at 200°C; (e) MET-pIRIR at 250 °C. All the data points were fitted using equation (3) and the fitting parameters were shown in each figure. All curves were normalized to the initial value.

Figure 3: Simulated isothermal decay at 20°C for the 50 °C IRSL (full curve) and 250 °C MET-pIRIR (dashed curve) signals.

Figure 4: Simulated isothermal decay at 20°C for IRSL at 50 °C (a) and MET-pIRIR 250 °C signals (b) after different preheat (PH) temperatures for 10 s, respectively.

Figure 5: Variation of D_e with preheat temperature for sample SM1. From three to six aliquots were measured at each preheat temperature, and the standard error is shown on each point. A preheat of 10 s at each temperature was applied prior to the natural, regenerative and test doses (Blair et al., 2005).

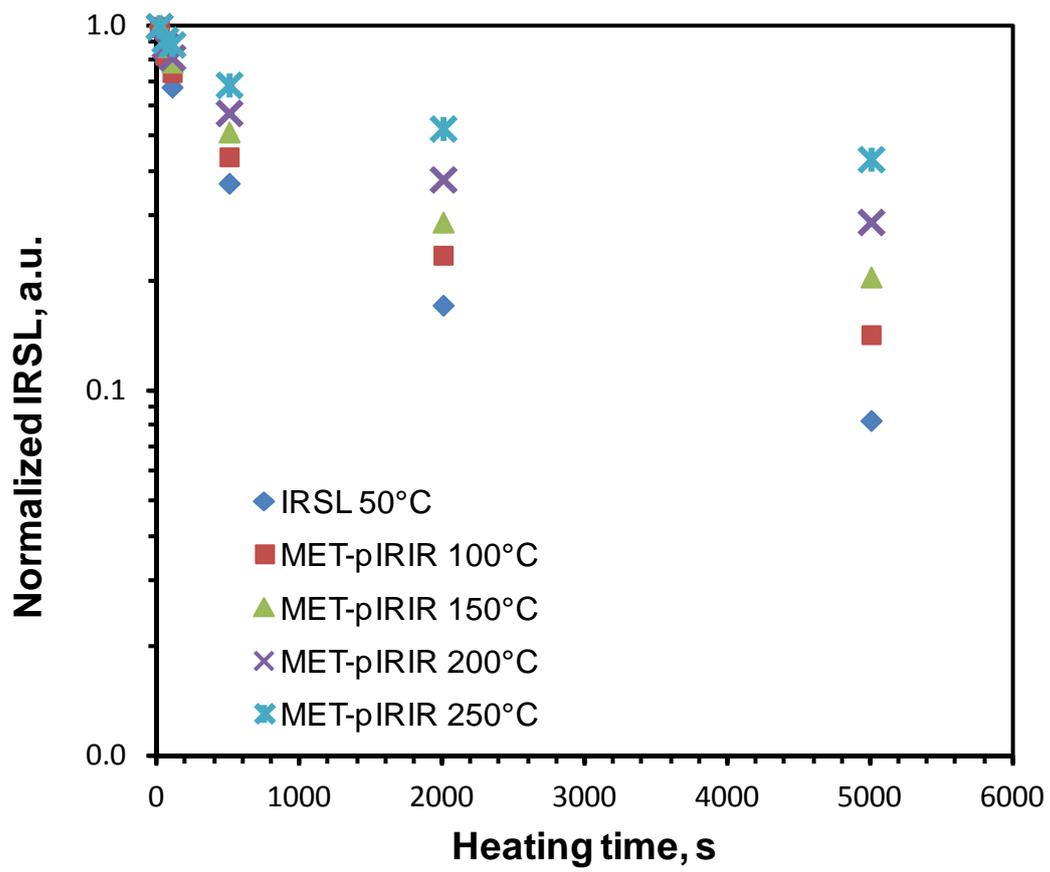


Fig. 1

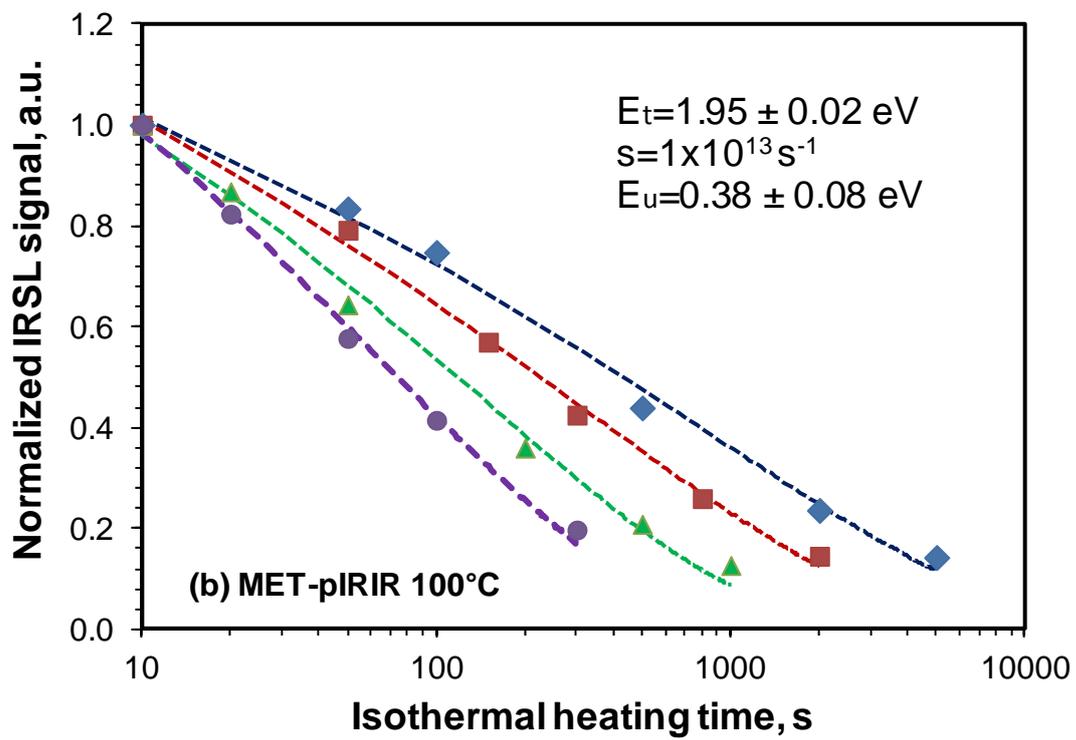
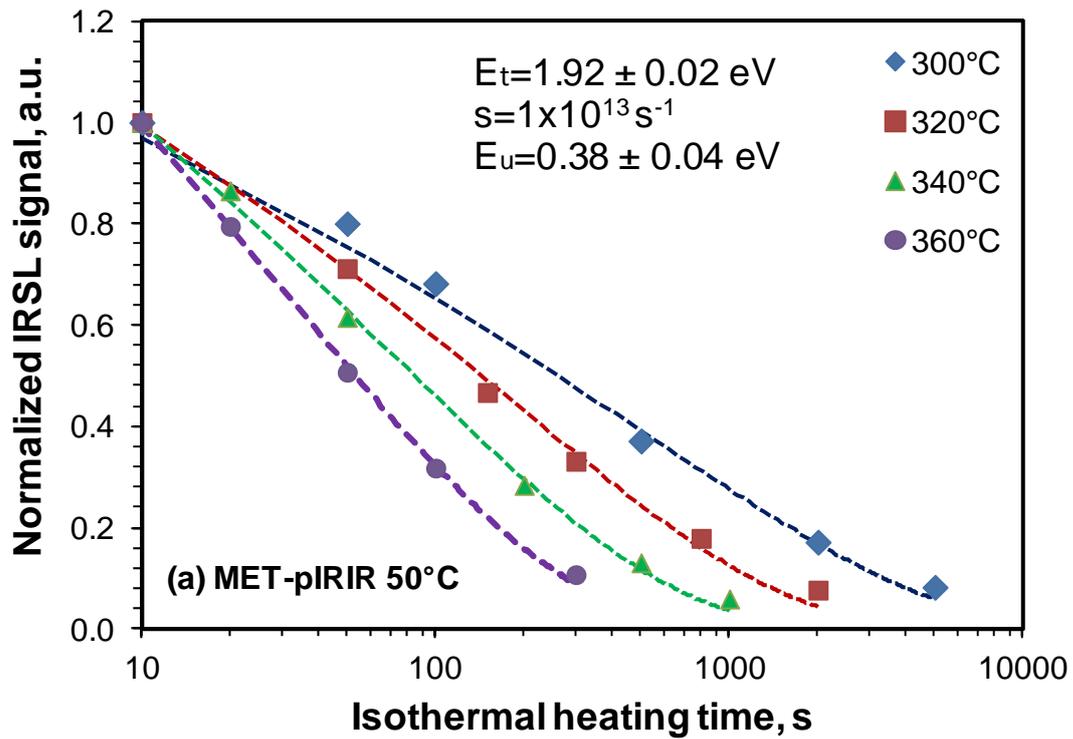


Fig. 2 (a) and (b)

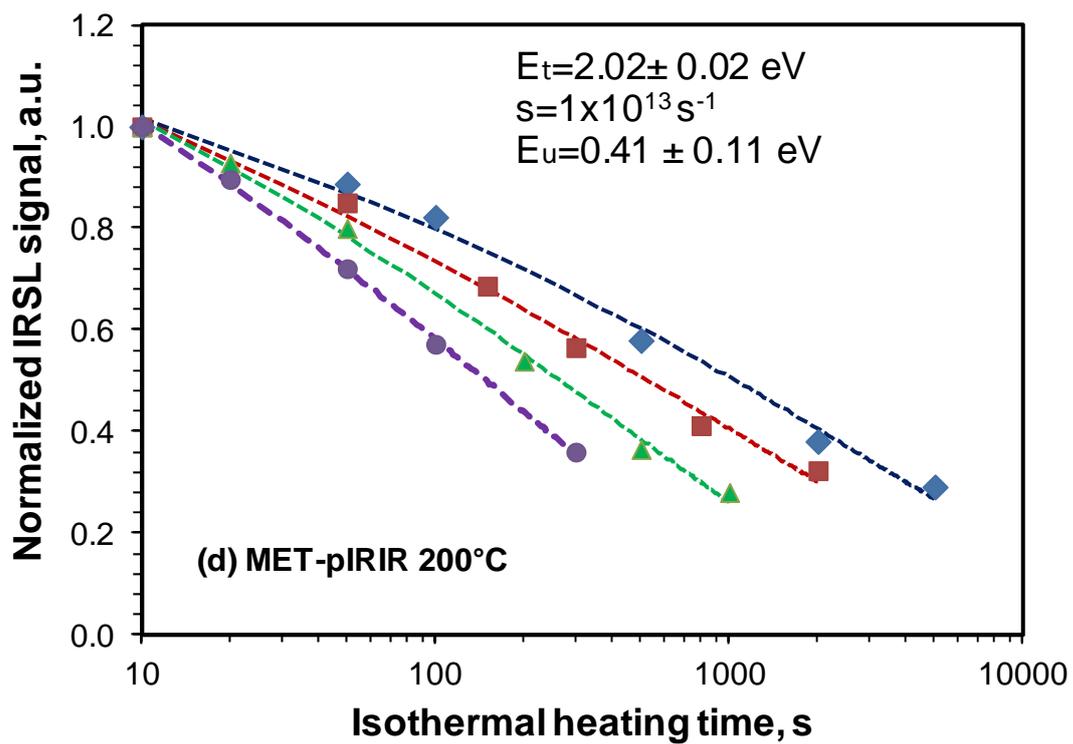
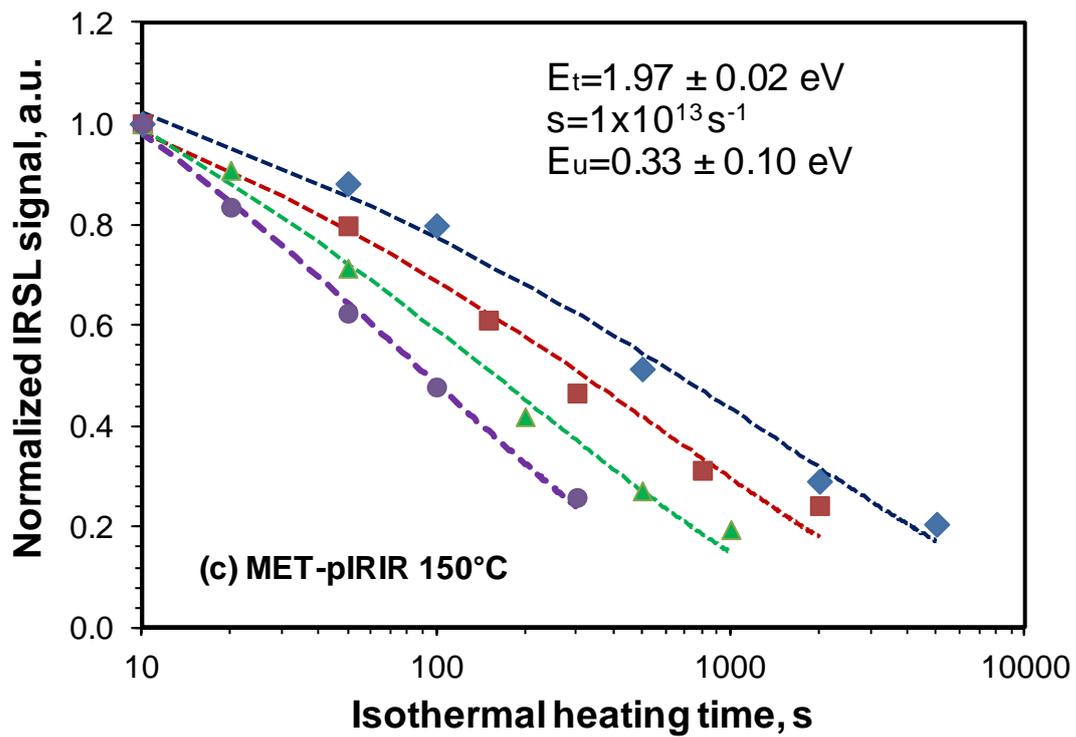


Fig. 2 (c) and (d)

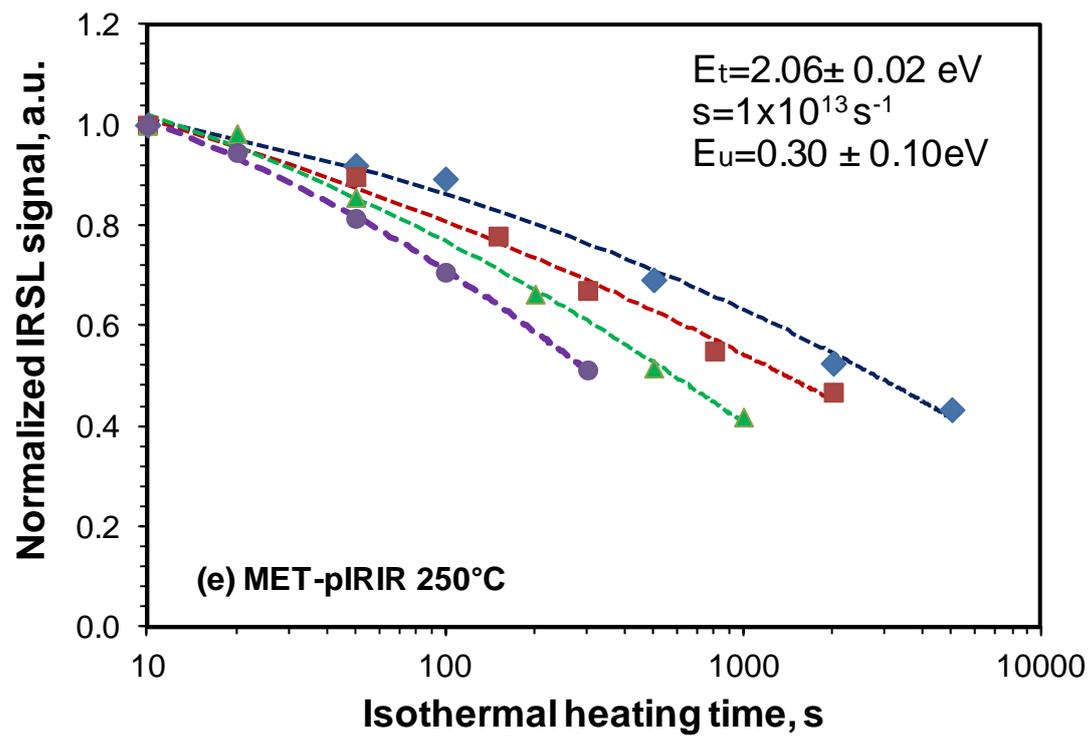


Fig. 2 (e)

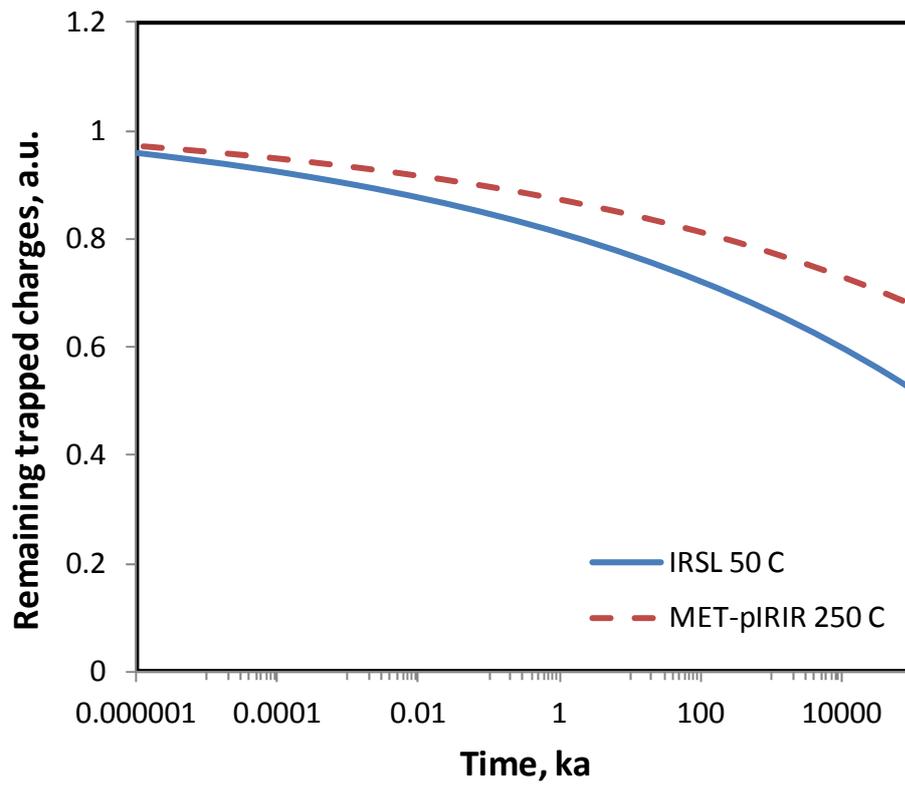


Fig. 3

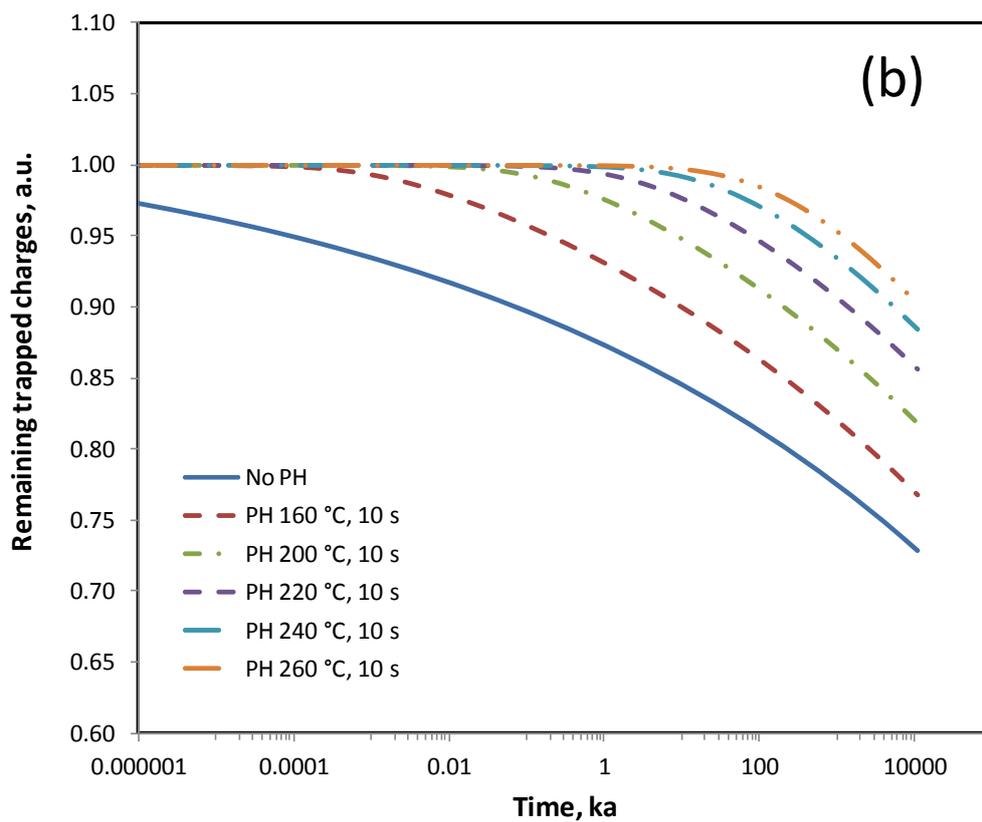
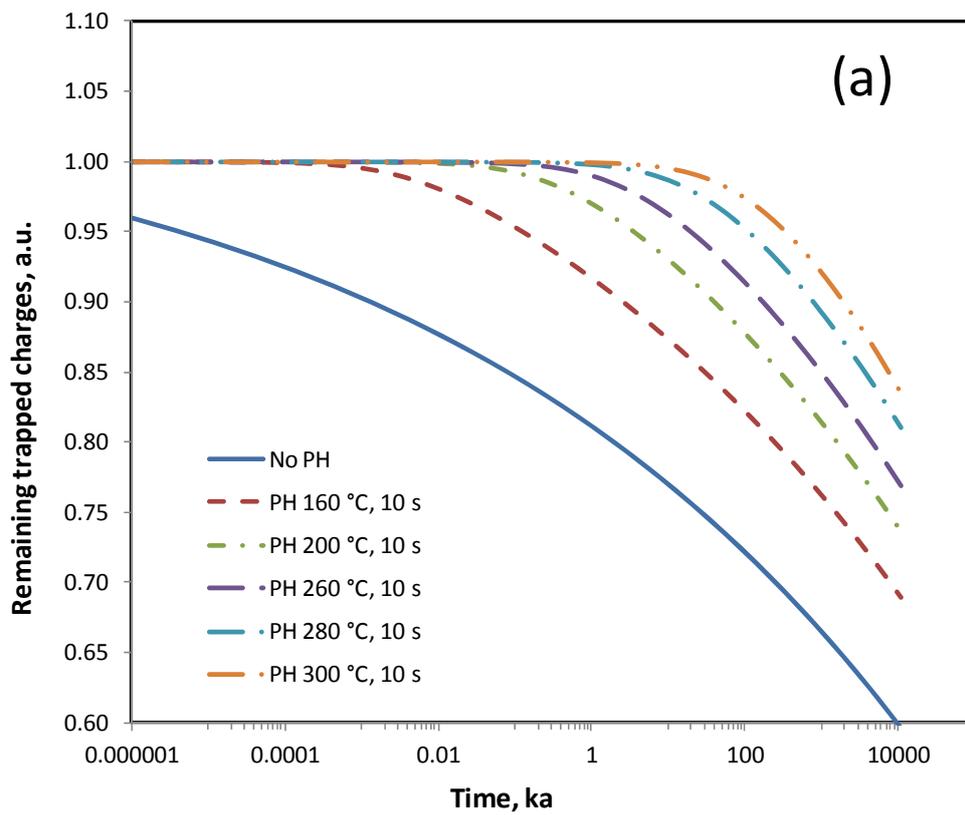


Fig. 4.

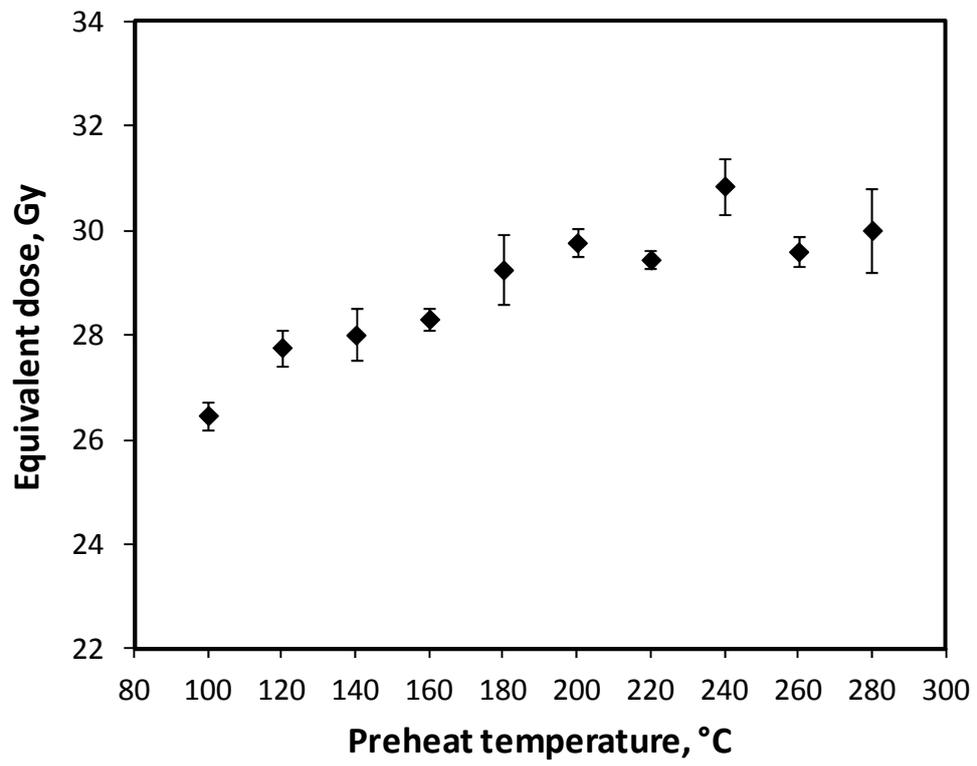


Fig. 5