

Thermoluminescence Response of LiF:Mg,Ti (TLD-100) Dosimeters After Shortened Annealing Profiles Under Megavoltage Photon Irradiation

Chong Yu Tong¹, Gunalan Ramachandran¹, Siti Hajar Zuber¹, Abdul Khaliq Mohd Saparudin¹, Siti Farizwana Mohd Ridzwan², Ung Ngie Min³, Ahmad Bazlie Abdul Kadir⁴, Nur Nadiah Mohd Rais⁵, Muhammad Safwan Ahmad Fadzil^{1,*}

¹*Diagnostic Imaging and Radiotherapy Program, Faculty of Health Sciences
Universiti Kebangsaan Malaysia, Jalan Raja Muda Abdul Aziz, 50300 Kuala Lumpur, Malaysia*

²*Department of Radiology, Hospital Canselor Tuanku Muhriz, 5600 Cheras, Kuala Lumpur,
Malaysia*

³*Clinical Oncology Unit, Faculty of Medicine, University of Malaya, 50603 Kuala Lumpur,
Malaysia*

⁴*Secondary Standard Dosimetry Laboratory, Malaysian Nuclear Agency, Bangi, 43000 Kajang,
Selangor, Malaysia*

⁵*Department of Diagnostic Imaging and Radiotherapy, Kulliyyah of Allied Health Sciences,
International Islamic University Malaysia, Jalan Sultan Ahmad Shah, Bandar Indera Mahkota,
25200, Kuantan, Pahang, Malaysia*

**Corresponding author (email: safwanfadzil@ukm.edu.my)*

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ABSTRACT

TLD-100 dosimeter is widely used in radiation dosimetry due to its near-tissue-equivalent response and reliable dosimetric properties. However, the manufacturer-recommended annealing protocol requires approximately 25 h, which may reduce operational efficiency in clinical and research settings. This study investigated the effects of modified annealing profiles on the thermoluminescence (TL) response and dosimetric performance of TLD-100. Six modified annealing profiles were investigated, comprising shortened heating durations at 400°C (45, 30 and 15 min) and shortened cooling durations at 80°C (12, 6 and 3 h). Following annealing, TLD-100 chips were irradiated using 6 MV photon beams at absorbed doses of 1, 5 and 10 Gy. TL responses and glow-curve characteristics were evaluated using a Harshaw 3500 TL reader. Percentage relative differences (PRDs) were used to assess variations in TL output relative to the reference annealing condition. Reducing the heating duration below 60 min increased TL output, with peak intensity increases significantly and PRD > 20% at some dose levels. These changes were accompanied by increased peak height, while the main glow-peak position remained within the observed temperature range across the investigated heating profiles. In contrast, reducing the cooling duration from 24 h to 3 h resulted in PRD of ≤ 5% for all investigated doses, with no substantial changes in glow-curve characteristics. Under the experimental conditions investigated, a 60 min heating stage

at 400°C appears necessary to achieve a reproducible TL response from TLD-100, whereas the cooling stage may be reduced without observable deterioration in dosimetric performance. These findings suggest that optimization of the annealing protocol may substantially improve operational efficiency without compromising TL stability.

Keywords: TLD-100; annealing protocol; photon beams; glow curve; trap parameters

INTRODUCTION

Accurate radiation dosimetry is essential for ensuring the safety and effectiveness of radiotherapy, diagnostic radiology and radiation protection. Even small deviations in delivered dose can affect treatment outcomes and patient safety [1-3]. This need for accurate dose measurement has become increasingly important with the use of modern radiotherapy techniques, which involve steep dose gradients and highly conformal dose distributions. Among the available dosimetric systems, thermoluminescence (TL) dosimetry remains widely used because of its high sensitivity, broad dynamic dose range, good reproducibility and relatively low cost [4,5].

Lithium fluoride doped with magnesium and titanium (LiF:Mg,Ti; TLD-100) is one of the most widely used TL dosimeters because of its near-tissue-equivalent effective atomic number and well-established dosimetric characteristics [6-8]. It is commonly used for patient dose verification, in vivo dosimetry and quality assurance of radiation treatment delivery systems. Its established performance has also made TLD-100 an important reference material for evaluating newer thermoluminescent and optically stimulated luminescence dosimeters [9]. Therefore, maintaining a stable and reproducible TL response is essential, as variations in dosimeter response can result in inaccurate dose estimation and potentially affect clinical decision-making.

The TL response of TLD-100 is based on the trapping and subsequent release of charge carriers at defect sites within the crystal. This process is influenced by the thermal history of the dosimeter. Consequently, pre-irradiation annealing is an important step in TL dosimetry because it removes residual trapped charge and restores the dosimeter to a reproducible baseline condition [10,11]. The manufacturer-recommended annealing protocol consists of heating at 400°C for 60 min, followed by cooling at 80°C for 24 h [12]. Although this protocol provides consistent dosimetric performance, its relatively long duration may be impractical in clinical and laboratory settings where rapid dosimeter preparation and turnaround are required. This limitation is particularly relevant in high-throughput dosimetry programs involving large numbers of dosimeters that require repeated annealing and irradiation.

Previous studies have demonstrated that changes in annealing conditions can affect TL intensity, glow-curve characteristics and signal stability by altering the distribution and occupancy of electron traps [13,14,15]. Inadequate annealing may result in incomplete trap emptying, leading to changes in dosimeter sensitivity and reduced reproducibility over repeated use. Recent studies have also reported that variations in heating conditions can significantly affect peak intensity and overall TL output [16, 17]. However, relatively few studies have systematically evaluated shortened annealing profiles, particularly with respect to their effects on TL stability following megavoltage photon irradiation.

This knowledge gap limits the development of more efficient annealing procedures that can reduce processing time without compromising dosimetric reliability. Therefore, this study investigated the effect of different annealing durations on the TL response of TLD-100 following irradiation with a megavoltage photon beam. Specifically, the study evaluated whether shortening the heating or cooling stages altered the measured peak TL response and glow-curve characteristics relative to the standard annealing condition. The objective was to characterize the response differences associated with shortened annealing conditions under the experimental parameters investigated.

MATERIALS AND METHODS

Sample Preparation

Commercial TLD-100 chips with nominal dimensions of $3 \times 3 \times 1$ mm³ were used in this study (Figure 1). Prior to experimentation, chips underwent a sensitivity screening using a uniform 1 Gy test dose. Individual Element Correction Factors (ECFs) were calculated to normalize the response of each chip to the batch mean. The initial sensitivity spread was within $\pm 5\%$. All dosimeters were handled using vacuum tweezers to minimize surface contamination, mechanical abrasion and inadvertent transfer of residues [7] that could affect the thermoluminescence (TL) response and measurement reproducibility. Prior to experimental use, each chip was visually inspected for surface contamination, discoloration, or other visible physical defects. Dosimeters exhibiting any of these characteristics were excluded from subsequent measurements.

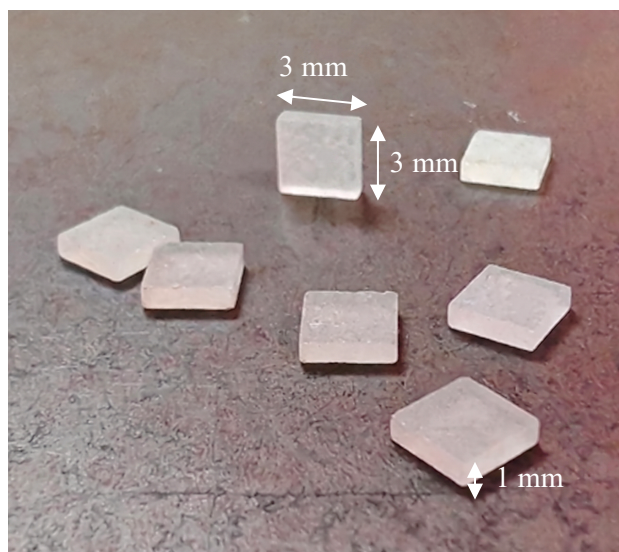


Figure 1. Geometry and dimensions of the commercially available LiF:Mg,Ti chips

For statistical assessment of the effect of annealing conditions, five independent TLD-100 chips were allocated to each annealing profile and dose condition ($n = 5$). The individual chips within each experimental group were treated as independent measurement units. For each condition, the mean peak TL intensity and standard deviation (SD) were calculated from the five individual chip measurements. Where ratios

relative to the standard annealing condition were reported, the uncertainty associated with the ratio was calculated from the corresponding variability of the modified and reference measurements.

Each group of TLD-100 was placed in a separately labelled capsule to maintain sample identity throughout the annealing, irradiation and TL readout procedures. All annealing treatments were performed using a TLD furnace LAB-01/400 (Thermo Fisher Scientific, Waltham, MA, USA). The oven was operated under controlled conditions with a temperature uniformity of $\pm 3^{\circ}\text{C}$ across the annealing area. The same oven, temperature setting and sample positioning protocol were maintained for all experimental groups to minimize variability associated with thermal gradients and equipment-related fluctuations.

Annealing Protocol

A standard annealing profile of 400°C for 60 min (heating) followed by 80°C for 24 h (cooling) was used as the reference condition, consistent with the conventional annealing protocol recommended for TLD-100 chips [12]. To evaluate the influence of annealing duration on TL response while eliminating temperature as a confounding variable, the heating and cooling temperatures were held constant at 400°C and 80°C , respectively, across all profiles; only the duration of the heating or cooling stage was varied.

Six manipulated annealing profiles were derived from the standard protocol and are summarized in Table 1. Three profiles (Heating A, B and C) investigated the effect of shortened heating duration 45, 30 and 15 min, respectively, while the cooling duration was kept at the standard 24 h. The remaining three profiles (Cooling A, B and C) investigated the effect of shortened cooling duration 12, 6 and 3 h, respectively, while the heating duration was kept at the standard 60 min. This design allowed the independent effects of heating-time and cooling-time reduction on TL sensitivity and stability to be evaluated separately, rather than as a combined or confounded duration effect.

Table 1. Standard and modified annealing profiles applied to TLD-100 chips, with shortened heating or cooling durations at fixed temperatures

	Heating temperature ($^{\circ}\text{C}$)	Time (min)	Cooling temperature ($^{\circ}\text{C}$)	Time (h)
Standard	400	60	80	24
Heating A	400	45	80	24
Heating B	400	30	80	24
Heating C	400	15	80	24
Cooling A	400	60	80	12
Cooling B	400	60	80	6
Cooling C	400	60	80	3

Following the cooling stage of each profile, the TLD chips were immediately transferred to a black, light-tight container to minimize exposure to ambient light and prevent premature loss of the induced TL signal prior to irradiation and readout. Both the annealing-to-irradiation and irradiation-to-readout intervals were standardized to 24 h

across all groups.

Irradiation Procedure

Annealed TLD-100 chips were irradiated using a 6 MV photon beam generated by an Elekta Versa HD linear accelerator (Elekta AB, Stockholm, Sweden) at the University Malaya Medical Centre. Linac output was verified prior to irradiation using a calibrated Farmer-type ionization chamber according to TRS-398 protocols. Three absorbed dose levels of 1, 5 and 10 Gy were delivered using a field size of 10 cm × 10 cm at a source-to-surface distance (SSD) of 100 cm. To establish charged particle equilibrium (CPE), the TLD capsules were placed on a 10 cm-thick solid water backscatter base (comprising 2 cm slabs) and covered with a 1.5 cm bolus, positioning the chips at the exact depth of maximum dose (d_{max}) for the 6 MV beam (Figure 2). The overall phantom arrangement was designed to approximate clinical beam conditions and to promote reproducible, uniform dose distribution across all irradiated samples.



Figure 2. The irradiation setup showing the arrangement of TLD-100 chips, solid water phantom slabs, bolus material and 6 MV photon beam from the linear accelerator

Readout Analysis

TL readout was carried out at the Dosimetry Laboratory, Universiti Kebangsaan Malaysia, using a Harshaw 3500 TLD reader (Thermo Fisher Scientific Inc., U.S.A.). Prior to readout, nitrogen gas was purged into the reader chamber (30 psig) to establish an oxygen-free environment, thereby suppressing triboluminescence effects. The readout time-temperature profile consisted of three stages: a 50°C preheat to eliminate unstable low-temperature peaks, a readout stage up to 300°C (at 10°C s⁻¹) for signal acquisition and a final 300°C post-read anneal to clear residual signals. Background signal was determined using un-irradiated control chips and subtracted from all raw readings. Reader stability was monitored using reference light constancy checks before each readout session to ensure small cooling-arm differences were not artifacts of reader fluctuation.

Glow curves of the TLD-100 chips were acquired using the Windows-based

Radiation Evaluation and Management System (WinREMS) software, which recorded the TL signal as a function of temperature and enabled identification of the temperature corresponding to maximum peak intensity (T_{max}), a key parameter in TL analysis. Raw glow curve data extracted from the TLD reader were subsequently exported to Microsoft Excel for further processing and graphical analysis. This allowed detailed examination of glow curve characteristics, including peak height, shape, symmetry and position, which provide insight into the thermal stability and kinetic behavior of the TL material. Peak height was utilized as the primary dosimetric signal rather than integrated area, as it provides a direct measure of the main dosimetric peak without requiring complex glow-curve deconvolution to isolate it from overlapping high-temperature structures.

The analysis was primarily descriptive, based on the percentage relative deviation (PRD), calculated using the following expression:

$$PRD (\%) = \left| \frac{I - I_0}{I_0} \right| \times 100$$

where I represents the mean peak TL intensity of a modified annealing profile and I_0 represents that of the standard annealing profile at the same dose level. The absolute value was used so that PRD represented the magnitude of deviation irrespective of whether the modified condition produced an increase or decrease in TL response. Comparisons between standard and modified annealing profiles were made by evaluating PRD values and by qualitative comparison of glow curve shape, peak position and peak width.

RESULTS AND DISCUSSION

Effect of Heating Duration on TL Response

Shortened heating durations produced systematically higher TL intensities across all dose levels, with the peak intensity ratio increasing progressively as heating time was reduced from 60 to 15 min (Table 2). Heating C (15 min) showed the greatest enhancement, followed by Heating B and Heating A. Correspondingly, PRD rose with shorter heating times, reaching values above 20% at 5 and 10 Gy. PRD was consistently highest at 5 Gy rather than 10 Gy, suggesting the effect of heating duration on TL response is not purely dose-dependent. PRD values for Heating B and C exceeded the predefined 5% comparison criterion at all doses, while Heating A remained within tolerance only at 1 Gy.

These results indicate that insufficient heating duration suggests incomplete thermal erasure of electron traps, which may allow residual charge carriers to persist and artificially elevate the TL signal in subsequent readings [18]. The 5% tolerance was adopted as a practical benchmark for assessing dosimetric variation relative to the standard annealing condition, rather than as a formal criterion of clinical accuracy or equivalence. Still, the consistent exceedance of this tolerance for heating durations of 30 min or less suggests that shortened annealing protocols, though attractive for reducing turnaround time, may compromise dosimetric accuracy.

Table 2. Normalized TL response metrics for shortened heating profiles at various doses, with peak intensity ratios calculated relative to the standard annealing protocol

Annealing profile	Heating time (min)	1 Gy			5 Gy			10 Gy		
		Mean peak intensity $\pm$ SD	(I/I ₀)	PRD (%)	Mean peak intensity $\pm$ SD	(I/I ₀)	PRD (%)	Mean peak intensity $\pm$ SD	(I/I ₀)	PRD (%)
Standard	60	3.61E+06 $\pm$ 1.48E+05	1.00	0.00	1.98E+07 $\pm$ 2.62E+06	1.00	0.00	4.65E+07 $\pm$ 2.56E+06	1.00	0.00
Heating A	45	3.75E+06 $\pm$ 1.15E+05	1.04	4.06	2.18E+07 $\pm$ 1.29E+06	1.10	9.99	5.05E+07 $\pm$ 4.43E+06	1.09	8.67
Heating B	30	3.88E+06 $\pm$ 1.42E+05	1.08	7.67	2.30E+07 $\pm$ 2.98E+06	1.16	15.85	5.24E+07 $\pm$ 3.60E+06	1.13	12.86
Heating C	15	4.22E+06 $\pm$ 1.97E+05	1.17	17.06	2.55E+07 $\pm$ 3.13E+06	1.29	28.84	5.69E+07 $\pm$ 5.01E+06	1.22	22.45

SD = Standard deviation; I/I₀ = Ratio of peak TL intensity; PRD = Percentage relative deviation

From a thermoluminescence perspective, the increased peak intensity observed following shortened heating durations may be associated with incomplete thermal emptying or redistribution of trapped charge during the annealing process. However, the present measurements do not permit the specific contribution of deep versus shallow trapping centers to be distinguished. No trap-kinetic analysis, glow-curve deconvolution, or independent measurement of residual trap occupancy was performed. Therefore, the observed increase in peak intensity should be interpreted primarily as an experimental response associated with shortened heating rather than as direct evidence of a specific trap population. The possibility that residual charge contributes to the increased TL signal is consistent with previous reports [10,17-19], but remains a proposed mechanism under the conditions investigated.

Glow Curve Characteristics under Heating Variation

Figure 3(a) – (c) show the glow curves of TLD-100 chips irradiated to 1, 5 and 10 Gy under the standard and three shortened annealing profiles. At all three doses, the curves kept the typical TLD-100 shape, with a main dosimetric peak at channel 100–115 and a smaller secondary peak near channel 150. The peak position and T_{max} remained within the observed range of 232–242°C across the investigated annealing conditions. These observations indicate that no substantial shift in the measured main glow-peak temperature was detected under the present experimental conditions; however, they do not by themselves establish that the underlying trap structure or trap population remained unchanged.

In contrast, main-peak intensity varied systematically with annealing duration: at all doses, Heating C (15 min) gave the highest signal, followed by Heating B, Heating A and the Standard profile, which was consistently lowest. This separation was most visible on the ascending side of the main peak, just before the peak, while the descending side remained tightly overlapping. A similar but weaker pattern appeared in the secondary

peak, especially at 5 and 10 Gy and the gap between profiles widened with dose, consistent with the higher PRD values at higher doses.

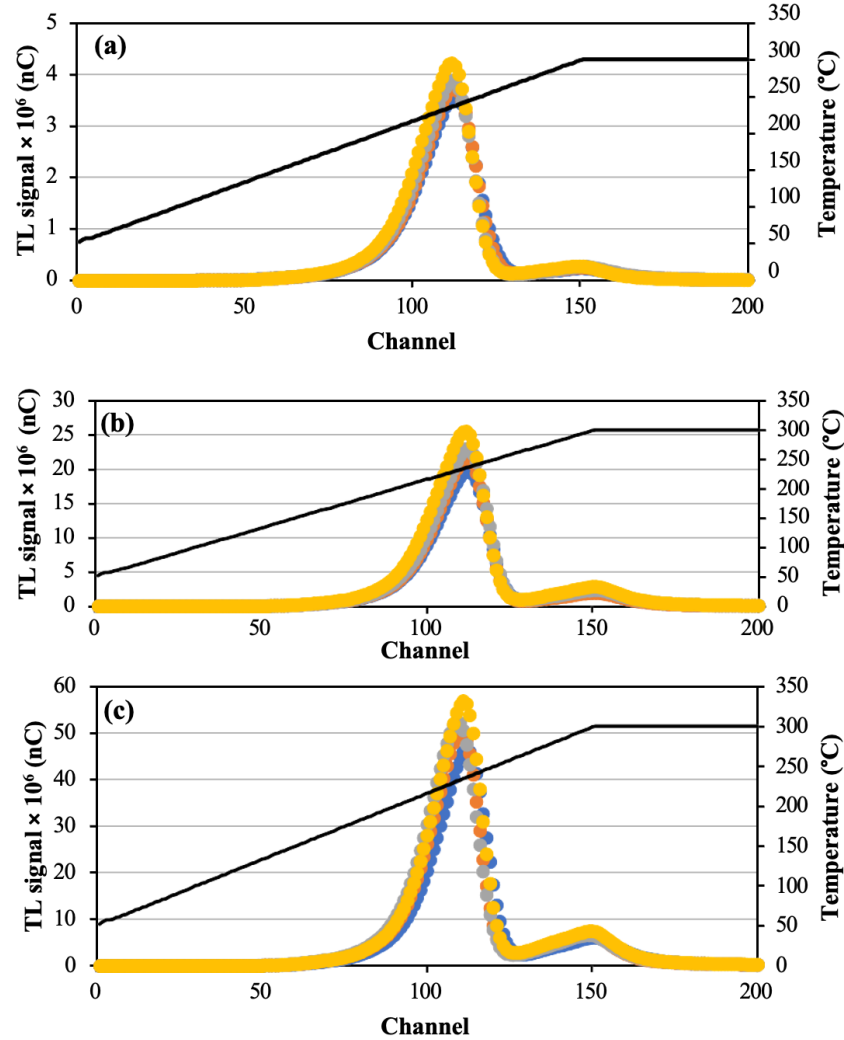


Figure 3. Glow curves of TLD-100 chips irradiated to (a) 1 Gy, (b) 5 Gy and (c) 10 Gy after standard and shortened heating profiles

Shortening the heating duration did not produce an evident shift in the main glow-peak position, as indicated by the stable T_{max} and main peak position. Instead, it mainly affects the amount of TL signal released from the main peak, which is represented by the peak magnitude. One possible explanation is that shorter annealing may not completely empty the shallow, low-temperature traps before the next irradiation and readout cycle [20]. During readout, residual charge from these traps may contribute to the signal on the ascending side of the main peak as the crystal is heated beyond T_{max} , increasing the peak intensity without shifting its position. This may also explain the observed dose dependence. As trap filling increases with radiation dose, the contribution of residual charge from incomplete annealing may become greater, resulting in larger differences in TL response at 5 and 10 Gy. In addition, differences in heating conditions during readout may slightly improve the efficiency of light emission near T_{max} , further increasing peak intensity without affecting the peak position.

Effect of Cooling Duration on TL Response

In contrast with heating duration, variation of cooling duration showed minimal effect on TL intensity and glow curve characteristics. TL intensity ratios for cooling profiles remained close to unity (0.96–0.99) and all PRD values were within $\pm 5\%$ across all doses (Table 3). At 1 and 10 Gy, PRD increased slightly as cooling time was reduced from 12 to 3 h, with Cooling C showing the largest deviation at both doses (3.03% and 3.61%). At 5 Gy, however, this trend did not hold. Cooling A produced the highest PRD (4.37%), while Cooling C showed almost no difference from standard (0.23%), indicating that the effect of cooling duration was less predictable at this dose.

Table 3. Normalized TL response metrics for shortened cooling profiles at various doses, with peak intensity ratios calculated relative to the standard annealing protocol

Annealing profile	Cooling time (h)	1 Gy			5 Gy			10 Gy		
		Mean peak intensity $\pm$ SD	I/I ₀	PRD (%)	Mean peak intensity $\pm$ SD	I/I ₀	PRD (%)	Mean peak intensity $\pm$ SD	I/I ₀	PRD (%)
Standard	24	3.61E+06 $\pm$ 1.48E+05	1.00	0.00	1.98E+07 $\pm$ 2.62E+06	1.00	0.00	4.65E+07 $\pm$ 2.56E+06	1.00	0.00
Cooling A	12	3.56E+06 $\pm$ 1.82E+05	0.99	1.30	1.90E+07 $\pm$ 3.04E+06	0.96	4.37	4.60E+07 $\pm$ 4.21E+06	0.99	1.14
Cooling B	6	3.54E+06 $\pm$ 2.26E+05	0.98	1.95	1.94E+07 $\pm$ 1.55E+06	0.98	1.75	4.51E+07 $\pm$ 3.97E+06	0.97	2.86
Cooling C	3	3.51E+06 $\pm$ 1.09E+05	0.97	3.03	1.97E+07 $\pm$ 3.23E+06	0.99	0.23	4.45E+07 $\pm$ 3.52E+06	0.96	3.61

SD = Standard deviation; I/I₀ = Ratio of peak TL intensity; PRD = Percentage relative deviation

Cooling duration appeared to have a less consistent effect on the TL response than heating duration. Since the cooling phase mainly allows the crystal and trapped charges to stabilize, rather than actively emptying or filling the traps [11,21], shortening this phase would be expected to have a smaller effect than shortening the heating phase. The fluctuation is likely attributable to statistical noise and baseline chip-to-chip variability rather than a physical consequence of the shortened cooling duration. The values are presented as mean peak intensity ratios obtained from five TLD chips per group. Overall, the observed chip-to-chip variation remained within the adopted $\pm 5\%$ dosimetric tolerance, indicating that the variations were within an acceptable range and did not suggest a meaningful effect of cooling duration on the TL response.

Glow Curve Characteristics under Cooling Variation

Figure 4(a)–(c) presents the glow curves of TLD-100 chips irradiated to 1, 5 and 10 Gy, respectively, under the standard and shortened cooling profiles. In contrast to the heating duration results, the glow curves for Standard, Cooling A, Cooling B and Cooling C showed near-complete superposition at all three doses, with no discernible shift in T_{max}

and no notable change in peak width, shape, or area under the curve. Both the main dosimetric peak and the secondary high-temperature structure remained consistent across all cooling durations, indicating that reducing the cooling period from 24 h to as little as 3 h had negligible effect on the TL response.

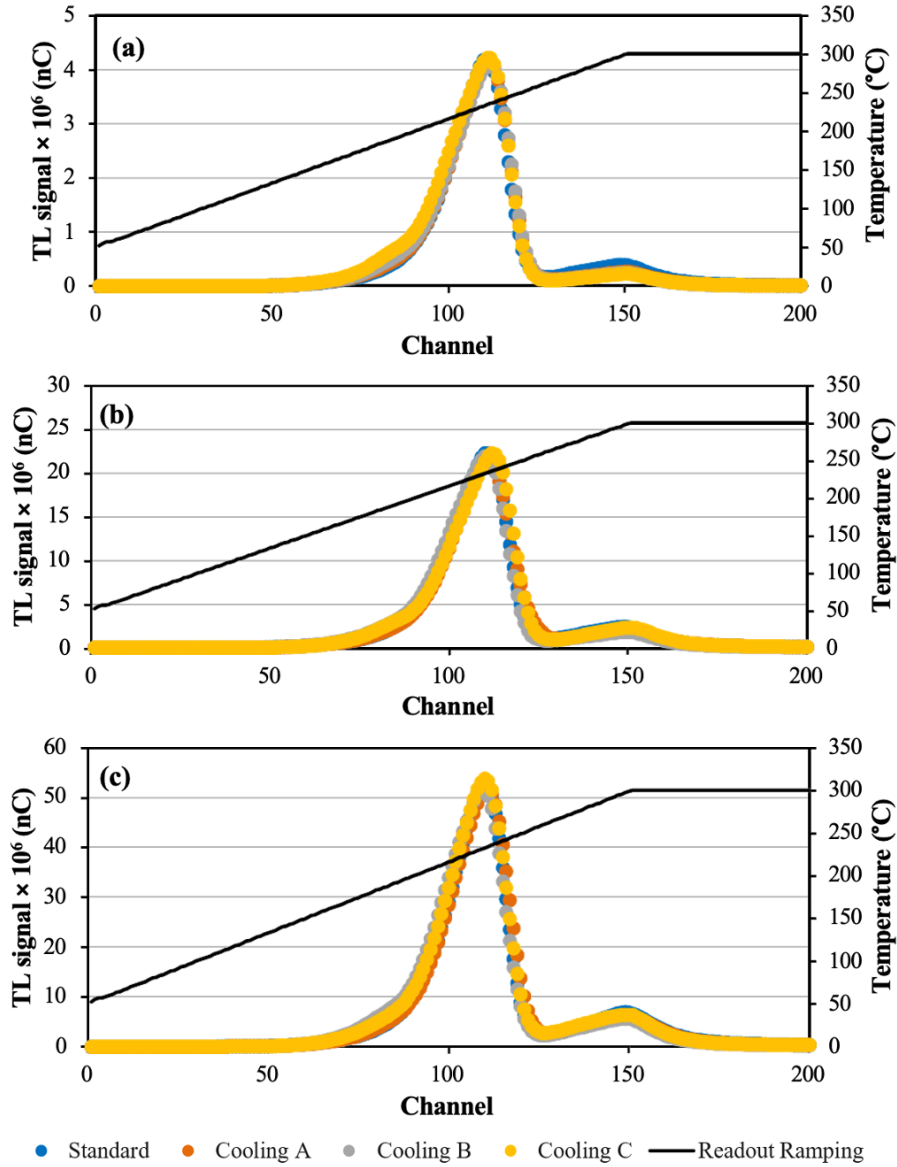


Figure 4. Glow curves of TLD-100 chips irradiated to (a) 1 Gy, (b) 5 Gy and (c) 10 Gy after standard and shortened cooling profiles

Notably, the area under the glow curve, which corresponds to the total trapped charge population released during readout [22], remained unchanged across all cooling durations. Since this parameter reflects the overall quantity of thermoluminescence signal rather than its temperature-dependent release profile, its invariance suggests that overall trap occupancy was likely not substantially altered by the shortened cooling periods, supporting the earlier observation that cooling primarily influences lattice

stabilization rather than trap population. This is consistent with previous reports identifying high-temperature annealing, rather than cooling rate, as the dominant factor governing TL trap emptying [23-25]. Accordingly, cooling durations as short as 3 h can be considered dosimetrically acceptable, remaining within the tolerance limits; however, this should not be interpreted as indicating 3 h to be the minimum or optimal duration, as shorter periods (e.g., 1 h or 30 min) were not investigated and may introduce lattice relaxation or residual thermal stress effects beyond the scope of the present study.

CONCLUSIONS

Under the experimental conditions investigated, shortened heating and cooling stages produced different effects on the measured TL peak response of LiF,Ti (TLD-100) dosimeters. Reducing the heating duration below the standard protocol consistently increased TL peak intensity and changed the magnitude of the peak. At several dose levels, the PRD exceeded the acceptable tolerance, indicating that the shortened heating duration may lead to incomplete emptying of deep electron traps. In contrast, reducing the cooling duration had minimal effect on the TL response. The PRD remained within the acceptable tolerance across all dose levels, with no substantial changes in glow-curve characteristics. This suggests that the cooling phase primarily facilitates stabilization of the crystal lattice without noticeably altering the overall trap occupancy. Overall, these findings indicate that the standard heating duration remains necessary to achieve a stable and reproducible TL response. However, the cooling duration can be substantially shortened without compromising dosimetric accuracy. This provides a practical opportunity to improve the efficiency of the annealing process in clinical and research dosimetry applications while maintaining measurement reliability.

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AUTHORS' CONTRIBUTIONS STATEMENT

The manuscript was written through the contributions of all authors. M.S.A.F., A.K.M.S. and U.N.M. conceptualized and designed the study and developed the methodology. C.Y.T. and G.R. conducted the formal analysis and investigation. C.Y.T. and M.S.A.F. prepared the original draft of the manuscript. M.S.A.F. and S.H.Z. contributed to the writing, review and editing of the manuscript. M.S.A.F., G.R. and A.K.M.S. were responsible for funding acquisition. S.H.Z., A.K.M.S., S.F.M.R., U.N.M., A.B.A.K., N.N.M.R. and M.S.A.F. supervised the work.

COMPETING INTERESTS

All authors declare that they have no competing interests that could have influenced the work reported in this paper.

DATA AVAILABILITY

The datasets used and/or analyzed during the current study are available from the corresponding author upon reasonable request.

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