THERMOLUMINESCENCE PROPERTIES OF K₂GdF₅:Tb MATERIAL IRRADIATED IN NEUTRON-GAMMA FIELDS OF ²⁴¹Am-Be SOURCE

by

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> Scientific paper https://doi.org/10.2298/NTRP2401037P

This paper presents the thermoluminescence properties of K_2GdF_5 :Tb material irradiated in a reference neutron-gamma field and its possibility to be used in neutron dosimetry. Double fluoride of K_2GdF_5 doped with 10 at% Tb³⁺ ions was synthesized under solid-state reaction conditions and irradiated in the reference neutron-gamma field of ²⁴¹Am-Be source. The thermoluminescence glow curves were measured by the Rexon UL-320 TLD reader connected with the WinRex-320 processing program for obtaining the integrated thermoluminescence signals (counts). The results show that the temperature peaks of the irradiated K_2GdF_5 :Tb material concerning the gamma and neutron fields at the distance of 100 cm from the source are at 230 °C and 300 °C, respectively. The sensitivity ratios of fast-to-thermal neutron and gamma-to-thermal neutron are 6.3 % and 18.2 %, respectively. The batch homogeneity and reproducibility are less than 30 % and 7.5 %, respectively. The linearity is less than 10 % in the dose range of less than 24 mSv. The detection threshold is less than 0.1 mSv, and the fading is 10.7 % after 90 days of irradiation. These thermoluminescence properties of K_2GdF_5 :Tb material considerably satisfy the requirements to be used in neutron dosimetry.

Key words: thermoluminescence properties, K_2GdF_5 : Tb material, neutron and gamma irradiation

INTRODUCTION

Recently, the development of thermoluminescence (TL) materials to be used in radiation detectors and solid-state dosimeters for various scientific and industrial applications has received increasing attention. There exists a commercial demand in TL phosphors for neutron dosimetry, measurement of neutron absorbed dose rates in space, and identifying the contribution of photons and neutrons to the total absorbed dose rate in neutron capture therapy [1-3]. Two isotopes of gadolinium, i. e., ¹⁵⁵Gd and ¹⁵⁷Gd, with high thermal neutron absorption cross-sections and relatively high contents in natural gadolinium, about 14.7 at% (atomic percentage), have the potential to be used in such TL materials. Nuclear reactions of the materials with neutrons in the energy range up to 10 MeV, *e.g.*, the neutron field of a ²⁴¹Am-Be, may emit prompt photons, internal conversion and Auger electrons, soft X-rays, and photons [4]. Though several Gd-based materials have been used for neutron detection as scintillators or imaging screen phosphors, no such material was proposed for neutron dosimetry as TL phosphors [1].

As known, several fluoride compounds doped with rare earth (RE) elements were found to be rather efficient TL phosphors [1]. Therefore, gadolinium fluorides doped with RE elements could be considered for the development of TL phosphors to be used in neutron detection. Previous works reported the synthesis of complex fluorides, e. g., K2GdF5, doped with RE ions, $e. g., Dy^{3+}$ and Tb^{3+} , based on the hydrothermal method and the TL properties of the materials irradiated to photon (X-ray, gamma) and neutron radiation [4-7]. Several studies in the synthesis of K2GdF5:Tb material in the powder form and their TL properties have been conducted. The synthesis of K₂GdF₅:Tb material using the hydrothermal method (at high temperature and high pressure) for investigating its TL properties under alpha, beta, and X-ray radiation was conducted by Hanh et al. [1]. The synthesis of the K₂GdF₅:Tb material using the solid-state reaction method was implemented by Vinh et al. [8-11]. Then, the TL properties of the K₂GdF₅:Tb material irradiated at high doses of beta, gamma, and neutron fields were investigated. The crystallographic structure by X-ray diffraction (XRD) and

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SEM, and the TL intensities of the K2GdF5: Tb material irradiated to the radiation fields indicated that the K₂GdF₅:Tb material with 10 at% Tb³⁺ ions had a good sensitivity at high radiation doses. However, the properties of the K₂GdF₅:Tb material irradiated to a reference gamma-neutron field at low doses (less than 20 mSv) have not been investigated. Besides, to promote the application of the material in personal neutron dosimetry, further studies need to be conducted to investigate the properties of the K₂GdF₅:Tb material, such as batch homogeneity, reproducibility, linearity, detection threshold, and fading, according to the International Organization for Standardization (ISO) 21909 [12] and International Electrotechnical Commission (IEC) [13, 14]. A comprehensive review of the development of neutron personal dosimetry can be found in [2]. One of the requirements for a dosimeter to be used for detecting neutron and gamma is the capability to discriminate between neutron and gamma components.

In the present work, the TL properties of the K_2GdF_5 :Tb material irradiated to fast, thermal neutrons and gamma in a reference neutron field at doses less than 20 mSv were investigated. The K_2GdF_5 :Tb material was synthesized based on the solid-state reaction method and irradiated in the reference ²⁴¹Am-Be field. The TL glow curves were measured using the Rexon UL-320 TLD reader. The TL properties such as batch homogeneity, reproducibility, linearity, detection threshold, and fading were evaluated and compared to the ISO and IEC.

MATERIALS AND METHODS

Synthesizing K₂GdF₅:Tb material

The initial material compounds consist of KF, GdF_3 , and TbF_3 supplied by Sigma-Aldrich Ltd. with a purity of 99.99 %. The K₂GdF₅ crystals doped with Tb^{3+} ions were synthesized based on the solid-state reaction method, according to the following chemical equation [8-11]

 $2KF + GdF_3 + Tb^{3+} \quad Tb^{3+}\text{-doped }K_2GdF_5$

Due to the water absorption of the KF compound, the material was dried at the temperature of 120 °C for 10 hours. Then, a mixture of KF, GdF₃, and TbF₃ with a mass ratio of 1:1.6622:0.1863 was ground in an agate mortar for 2 hours to ensure the mixture homogenized with small particles. The mixture was placed in a graphite boat and heated in an argon gas flux at the temperature of 620 °C for 6 days. After heating, the material was obtained as white hard pellets. It was crushed up to a particle size of about 50-100 m and washed with distilled water and ethanol several times. The material was then dried at the temperature of 120 °C for 10 hours, annealed at the temperature of 420 °C for one hour, and cooled to room temperature. Finally, the synthesized material in powder form was divided into samples with a weight of 20 mg and sealed in black plastic capsules for use as TL detectors (TLD). The synthesis procedure of the material was carried out at the Sample Treatment Laboratory of the Dalat Nuclear Research Institute (DNRI). The synthesis process of one batch of the K₂GdF₅:Tb material took about 9 days.

Irradiation and measurement of TL signals

The TLD were irradiated in a reference neutron field of the ²⁴¹Am-Be source at the Tertiary Standard Dosimetry Laboratory (TSDL) of DNRI. The emission rate of the source is 5.4094 10^6 s⁻¹ (6 %) in 2023. The fast neutron field was established with the source exposure in the air, while the thermal neutron field was established with the source moderated by the eleven polyethylene sheets with a total thickness of 11 cm. The neutron dose rates of the fast and thermal neutron fields at the irradiation positions were determined using a neutron spectrometer with the ³He detector and the PE bonner-cylinder system [15]. All measurements were conducted at the same irradiation position of 100 cm away from the source. After irradiation, the TLD were measured for the TL intensity (TL signals, unit in counts) using the Rexon UL-320 TLD reader connected with the WinRex-320 processing program. To optimize the measuring cycle, the setting parameters of the Rexon UL-320 TLD reader were optimally determined. The main setting parameters included the heating temperature range, temperature heating rate (possible values of $2 \degree Cs^{-1}$, $5 \degree Cs^{-1}$, $10 \degree Cs^{-1}$, $15 \degree Cs^{-1}$, and 20 °Cs⁻¹), heating type (linear or non-linear), and measuring cycle (total measuring steps). By changing the setting parameters, the optimal TL reading process was selected when a stable integrated TL intensity and a distinguished TL spectrum were obtained. The TL glow curves were then derived from the TL signals [16]. Figure 1 shows the reference neutron field of the ²⁴¹Am-Be source at DNRI.

To determine the TL properties of the K_2GdF_5 :Tb materials irradiated in the reference neutron field, the TLD were divided into several groups, each group consisting of ten TLD, to be irradiated at different doses. For each configuration, the sample group of ten TLD was irradiated simultaneously. Several procedures were implemented for irradiating the TLD in the thermal and fast neutron fields and measuring the integrated TL output signals (abbreviated to signal, in a unit of count) depending on the TL properties to be evaluated. In each procedure, a group of ten TLD of the same material batch was prepared for background measurement, *i. e.*, without irradiation.

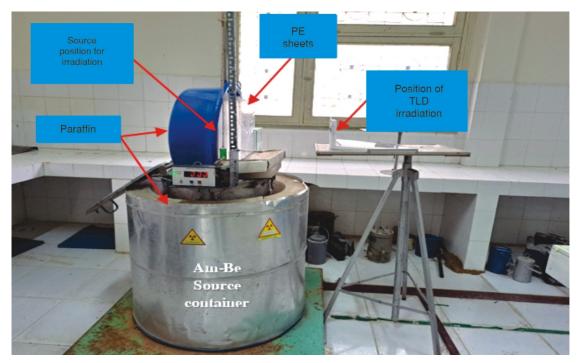


Figure 1. The reference neutron field of the ²⁴¹Am-Be source

Temperature peaks for gamma and neutron

Two groups of TLD were prepared and irradiated at the same dose of 8 mSv in the fast neutron field and the thermal neutron field, respectively. The signals (counts) of the irradiated TLD were then measured to determine average temperature peaks of gamma, thermal, and fast neutrons.

Sensitivity ratios of fast and thermal neutrons to gamma

Two groups of TLD were prepared and irradiated at the same dose of 1.7 mSv, one with the fast neutron field and the other with the thermal neutron field. The signals were measured to determine sensitivity ratios of fast, thermal neutron, and gamma fields.

Batch homogeneity

A group of ten TLD were irradiated at the same dose of 3 mSv in the thermal neutron field. The signals were measured to determine batch homogeneity.

Reproducibility for thermal neutron

A group of ten TLD was irradiated at the same thermal neutron dose of 10 mSv. The signals were then measured and recorded. The TLD were then annealed and irradiated again at the same dose of 10 mSv, and the signals will be determined again. This process was repeated ten times to evaluate reproducibility.

Linearity at low doses

Four groups of TLD were prepared and irradiated at the thermal neutron doses of 3.24 mSv, 5.23 mSv, 7.85 mSv, and 23.84 mSv, respectively. The signals were measured to determine a linear response.

The detection threshold of a dose

A group of ten TLD of the same material batch and storage (no irradiation). The signals were measured after 1 day and the second one after 30 days for the TLD to determine a detection threshold.

Fading

Seven groups of TLD of the same material batch were prepared and irradiated at the same thermal neutron dose of 3 mSv and stored. The signals were measured after the storage time of 1, 7, 14, 21, 30, 60, and 90 days, respectively, to determine a decrease in the signals with time.

RESULTS AND DISCUSSION

Temperature peaks for gamma and neutron

Figures 2 and 3 (taken from the screen of the Rexon UL-320 TLD reader) illustrate the typical TL glow curves of the TLD irradiated in the fast and thermal neutron fields, respectively. In these figures, the

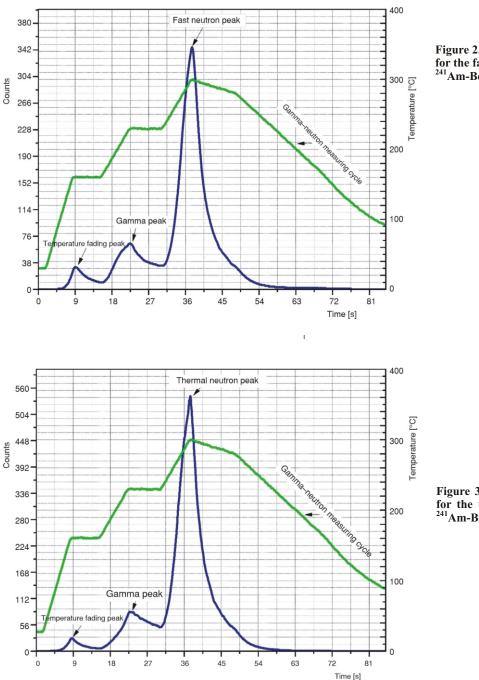


Figure 2. Typical TL glow curve for the fast neutron field of ²⁴¹Am-Be source

Figure 3. Typical TL glow curve for the thermal neutron field of ²⁴¹Am-Be source

blue lines are the glow TL curves (count versus measuring time) and the areas under these lines are the integrated TL signals (counts), and the moss-green lines are measuring cycles (temperature versus measuring time). To obtain the total counts at the temperature peaks in the TL glow curves, it was adjusted manually ROI (cursor) around the temperature peak according to the horizontal axis. The area was marked, and the total counts were read on the screen of the TL reader. The errors of the total counts for the peaks were less than 3 %. The neutron-gamma measuring cycle is an optimal choice for measuring the TL glow curves including neutron and gamma from ²⁴¹Am-Be source (the first node corresponds to the measuring time of 8 seconds from room temperature to 160 °C at the heating rate of 20 °Cs⁻¹; the second one corresponds to the time of 7 seconds at 160 °C; the third one corresponds to the time of 7 seconds from 160 °C to 230 °C at the heating rate of 10 °Cs⁻¹ for measuring gamma; the fourth one corresponds to the time of 8 seconds at 230 °C; the fifth one corresponds to the time of 7 seconds from 230 °C to 300 °C at the heating rate of 10 °Cs⁻¹ for measuring neutron. The next node corresponds to the total measuring time of 48 seconds to reduce temperature from 300 °C to 20 °C to make the TL distribution of neutron in the glow curve with a Gaussian shape).

One can see the simple shapes of the TL glow curves with three characteristic temperature peaks. The

Batch homogeneity (BH) is calculated as follows [12, 14]

BH
$$\frac{N_{max} N_{min}}{N_{min}}$$
 0.3 (1)

where N_{max} and N_{min} are the maximum and minimum counts, respectively. The value of BH should not exceed 30 %, according to the ISO and IEC [12, 14]. Table 2 summarizes the signals (counts, symbol as Ni) of the TLD of the same material batch irradiated at the thermal neutron dose of 3 mSv. The results show that the maximum and minimum counts are $N_{max} = 2607$ and $N_{min} =$ = 2110, respectively. Thus, the BH of 23.6 % is determined. This value is smaller than 30 %, *i. e.*, it satisfies the requirement for radiation dosimetry [12, 14].

Reproducibility in thermal neutron field

Batch homogeneity

Reproducibility (RE) is calculated from the following formula [12, 14]

$$RE \quad \frac{\overline{SD} \quad 1}{\overline{N}} \quad 0.075 \tag{2}$$

where \overline{SD} is the average standard deviation of *n* irradiations (*n* = 10 in this study), 1 is the confidence interval (95 %) of \overline{SD} obtained in [14], and \overline{N} – the average count of n irradiations. The value of RE should not exceed 7.5 % as recommended by the ISO and IEC [12, 14]. Table 3 summarizes the signals for ten times irradiation at the same thermal neutron dose of 10 mSv for the TLD. The RE of 7.4 % is obtained, which satisfies the basic requirement for radiation dosimetry [12, 14].

Linearity at low doses

Linear response (LR) is calculated from the following formula [12, 14]

$$0.9 \quad \text{LR} \quad \frac{\overline{N} \quad 1}{c} \quad 1.1 \tag{3}$$

Table 2. Counts of 10 TLD irradiated at the same the	rmal
neutron dose of 3 mSv	

Number of TLD	N _i (count)
1	2464
2	2236
3	2530
4	2114
5	2110
6	2447
7	2335
8	2607
9	2523
10	2274
BH [%]	23.6

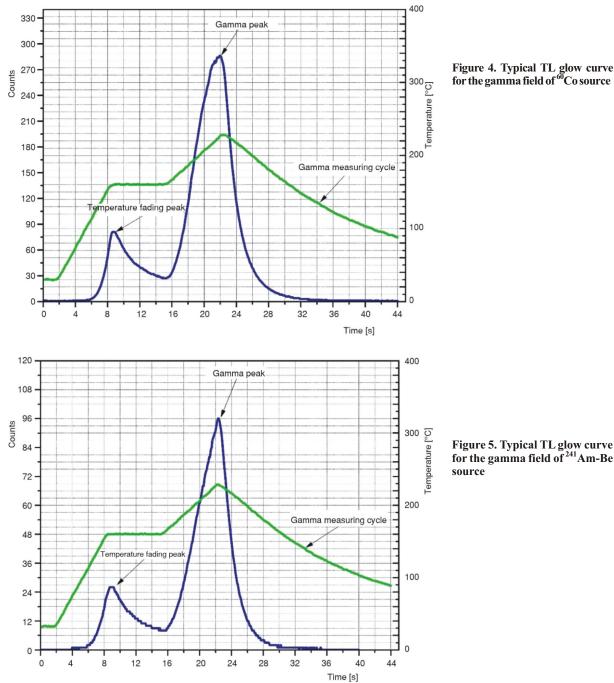
first peak on the left side at the temperature of 160 °C (at a heating rate of 20 °Cs⁻¹) corresponds to thermal fading, which is often quenched by the thermal fading effect and is not used in dosimetry. The second peak in the middle at 230 °C (at a heating rate of 10 °Cs⁻¹) corresponds to gamma. The third peak on the right side at 300 °C (at a heating rate of 10 °Cs⁻¹) corresponds to fast and thermal neutrons. To confirm the second peak corresponding to gamma in the ²⁴¹Am-Be field, the TLD were irradiated separately by photon sources (such as X-ray, ¹³⁷Cs, ⁶⁰Co), and the results show that the temperature peak concerning photon is at 230 °C. Figures 4 and 5 illustrate the TL glow curves of the TLD irradiated in the gamma fields of 60Co and ²⁴¹Am-Be sources, respectively. Here, the gamma measuring cycle was used to get the glow curves only in the range of temperatures for gamma. This affirmed that the temperature peaks at 230 °C and 300 °C are the dosimetric peaks of gamma and neutron, respectively. Compared to the results reported in [1] for the K₂GdF₅:Tb material synthesized by the hydrothermal method and the TL glow curves under X-ray irradiation, it is confirmed again that the temperature peak at 230 °C due to gamma irradiation in this study is consistent with that found in [1]. This measurement of the K_2GdF_5 doped with Tb³⁺ indicated the promising TL properties for detecting neutrons and separating its dose component in a gamma-neutron field. Since the measurements were performed with the optimal measuring cycle, the two peaks of gamma and neutron were separated clearly. This can be explained as the depth of gamma energy traps is less than that of the neutron, *i. e.*, the peak of gamma at a lower temperature than that of the neutron.

Sensitivity ratios of fast, thermal neutron, and gamma

The TL flow curves for fast neutron, thermal neutron, and gamma fields have been measured, evaluated, and summarised in tab. 1. Table 1 represents the total areas (average count, \overline{N}) and their standard deviations (\overline{SD}) under the corresponding TL peaks. The TLD were irradiated with a total dose of 8 mSv. This is because it gives sufficient statistical counts for the investigation within the dose range of less than 20 mSv, and the irradiation time was reasonably estimated to save the experimental cost. The sensitivity ratios on the intensities of fast-to-thermal neutron and gammato-thermal neutron are 6.3 % and 18.2 %, respectively. This indicates that the TLD are not sensitive to fast neutrons, but highly sensitive to gammas.

Table 1. Average counts of the TLD irradiated at the same dose of $8 \,\mathrm{mSv}$ in the gamma, fast, and thermal neutron fields

0			
Radiation field	\overline{N} (count)	(\overline{SD}) (count)	
Gamma	5013	87	
Fast neutron	1733	126	
Thermal neutron	27502	933	





Irradiation times	$\overline{N_i}$ (count)
1 st	11330
2 nd	12461
3 th	10324
4 th	11480
5 th	12646
6 th	12480
7 th	12391
8 th	12758
9 th	12102
10 th	11905
N	11988
SD	717
RE [%]	7.4

where \overline{N} is the average count for each group of TLD irradiated at the same dose, 1- the half-width of the confidence interval (95 %) of \overline{N} relative to the *i*th group of TLD obtained in [14], and C – the irradiated dose of the i^{th} group of TLD (in mSv). The value of LR should vary within the range of 0.9-1.1, which means the deviation of the LR should not be greater than 10 % over the irradiated dose range, according to the recommendation of ISO and IEC. Table 4 summarizes the signals (counts) for the four groups of TLD irradiated at the thermal neutron doses of 3.24 mSv, 5.23 mSv, 7.85 mSv, and 23.84 mSv, respectively. The LR values for the four groups of TLD are in the ranges of 0.99-1.07, 0.91-1.08, 0.92-1.05, and 0.93-1.08, respectively. The values satisfy the requirement for radiation dosimetry in

Table 4. Counts for four	groups of TLD irradiated at	
different thermal neutron	doses	

Number of TLD	3.24 mSv	5.23 mSv	7.85 mSv	23.84 mSv
1	4412	5223	5186	10152
2	4235	5672	6166	10130
3	4095	4145	5833	12066
4	4680	4526	6411	9438
5	4695	5310	5308	8074
6	4291	4173	4840	11202
7	4783	4365	5406	10677
8	4544	4985	5673	10742
9	4537	5097	5988	9606
10	4874	5697	5041	10158
N	4515	4914	5585	10225
LR	0.99-1.07	0.91-1.08	0.92-1.05	0.93-1.08

[12, 14]. Figure 6 depicts the linear response as a relationship between the signals (counts) and the thermal neutron doses (mSv). The response is linear with a good correlation coefficient ($R^2 = 0.998$). The discrepancies in the values of the doses are less than 10 %.

The detection threshold of the dose

The detection threshold (DT) of the dose was calculated by the following formula [12, 14]

$$DT \quad t_n \quad \text{SD} \quad \text{H}$$
 (4)

where t_n is the student's coefficient for *n*-1 DoF (n = 10 in this study). The value of $t_n = 2.26$ was taken in [14] and SD is the average standard deviation for the TLD. The value of DT should not exceed the value of H = 0.1 mSv as recommended by the ISO and IEC in [12, 14]. Table 5 summarizes the signals (counts, symbol as Ni) on 1st day and 30th one after irradiation. The detection threshold was determined as 1135 counts, corresponding to the dose of 0.095 mSv. This value satisfies the requirement for radiation dosimetry [12, 14].

Fading

The seven groups of TLD were irradiated at the same thermal neutron dose of 3 mSv and then measured at 1, 7, 14, 21, 30, 60, and 90 days after irradiation, respectively. The arithmetic means of the signals (counts) for the seven TLD groups were calculated and summarized in tab. 6. Figure 7 depicts the decreasing rate of the counts of the TLD with time after irradiation. As shown in fig. 7, the fading decreases fast from the first day to the 20th day and then decreases slowly after that time. On the 90th day after irradiation, the fading is 10.7 %. This value is approximately the requirement for radiation dosimetry as mentioned in [12, 14]. According to the ISO and IEC, the fading should be 10 % on the 90th day under standard test conditions (Ambient temperature from 10 °C to 22 °C, atmospheric pressure from 86 kPa to 106 kPa, relative humidity from 50 % to 65 %).

Table 5. Counts on 1st day and 30th day after irradiation

Number of TLD	N_i (count) on the 1 st day	N_i (count) on the 30 th day
1	1538	34036
2	1485	33763
3	1493	33563
4	1515	33669
5	1531	34057
6	1578	33167
7	1583	33510
8	1501	34434
9	1501	34767
10	1697	33351

Table 6. Counts with time after irradiation (fadi

TLD groups	Days after irradiation	\overline{N}_i (count)	Fading [%]
1	1	4109	0.0
2	7	3944	4.0
3	14	3764	8.4
4	21	3684	10.3
5	30	3680	10.4
6	60	3674	10.6
7	90	3671	10.7

CONCLUSIONS

The TL properties of the K2GdF5: Tb material irradiated in the reference neutron-gamma field were investigated for possible use in neutron dosimetry. The K2GdF5:Tb material was synthesized under the solid-state reaction conditions and irradiated in the reference neutron field of the ²⁴¹Am-Be source. The TL glow curves were measured using the Rexon UL-320 TLD reader to determine the TL properties, such as batch homogeneity, reproducibility, linearity, detection threshold, and fading. The results show that the temperature peaks for gamma and neutron fields are at 230 °C and 300 °C, respectively. The sensitivity ratios of fast-to-thermal neutron and gamma-to-thermal neutron are 6.3 % and 18.2 %, respectively. The batch homogeneity and reproducibility are less than 30 % and 7.5 %, respectively. The linearity is less than 10 % in range of thermal doses of less than 24 mSv. The detection threshold is smaller than 0.1 mSv, and the fading is 10.7 % at 90th day after irradiation. These TL properties considerably satisfy the basic requirements of the ISO and IEC for possible use in neutron dosimetry.

ACKNOWLEDGMENT

The authors are grateful to Dr. Xuan-Vinh Ha and Mrs. Phan-Thao-Tien Doan of NITRA for their valuable assistance in the material synthesis procedure and during the preparation of this revised manuscript.

This research was supported by the Ministry of Science and Technology, Vietnam 2022-2023 under Grant No. DTCB.03/22/VNCHN.

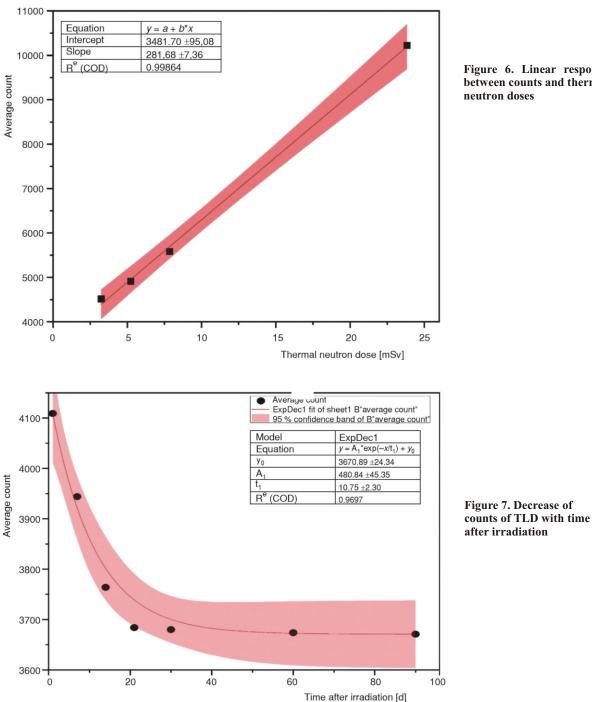


Figure 6. Linear response between counts and thermal neutron doses

AUTHORS' CONTRIBUTIONS

V. T. Phan, V. H. Nguyen, H. T. Pham, and V. D. Pham designed the study, performed the measurements, analyzed the data, and wrote the initial draft of the manuscript. D. K. Tran and H. N. Tran analyzed the data, wrote the initial draft, and reviewed and edited the final manuscript.

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Received on April 11, 2014 Accepted on June 24, 2024

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ТЕРМОЛУМИНЕСЦЕНТНА СВОЈСТВА К2GdF5:Тb МАТЕРИЈАЛА ОЗРАЧЕНОГ У НЕУТРОН-ГАМА ПОЉИМА ²⁴¹Аm-Ве ИЗВОРА

Приказана су термолуминисцентна својства K_2GdF_5 :Тb материјала озраченог у референтном неутрон-гама пољу и могућност његове употребе у неутронској дозиметрији. Двоструки флуорид K_2GdF_5 допиран са 10 at% Tb³⁺ јона синтетизован је у реакционим условима чврстог стања и озрачен у референтном неутрон-гама пољу извора ²⁴¹Am-Be. Криве термолуминисцентног сјаја мерене су Rexon UL-320 TLD читачем повезаним са програмом за обраду WinRex-320, за добијање интегрисаних сигнала термолуминисценције. Резултати показују да су температурни врхови озраченог K_2GdF_5 :Тb материјала који се односе на гама и неутронско поље на удаљености од 100 ст од извора, на 230 °C и 300 °C, респективно. Односи осетљивости брзих према термичким неутронима и гама према термичким неутронима су 6.3 % и 18.2 %, респективно. Хомогеност серије и поновљивост мање су од 30 % и 7.5 %, респективно. Линеарност је мања од 10 % у опсегу доза мањем од 24 mSv. Праг детекције је мањи од 0.1 mSv, а слабљење је 10.7 % после 90 дана зрачења. Ова својства термолуминисценције материјала K₂GdF₅:Tb у великој мери задовољавају захтеве за употребу у неутронској дозиметрији.

Кључне речи: шермолуминисценшна својсшва, K₂GdF₅:Тb машеријал, неушронско и гама зрачење