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TEMPERATURE-RELATED THERMOLUMINESCENCE INTERNAL PARAMETERS OF NANO-ALUMINUM

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Abstract: The basic thermoluminescence properties of irradiated nano-α-alumina particles are examined in this study, along with how they react to different heating speeds. TL luminescence curves are recorded for the investigation, and they show a clear peak with a maximum at about 202 °C. The peak continuously moves towards lower temperatures as dosage levels rise, demonstrating adherence to non-first-order kinetics (b^{1}1). XRD analysis was used to determine the crystallite size, which was determined to be 40 nm. Specimens were treated to a 6 kGy dosage in order to determine the kinetic parameters for nano α-$Al_2O_3$and investigate the effect of the heating rate on the TL glow curve. TL glow curves were then recorded using various heating speeds (2, 4, 6, 8, and 12 °C/s) throughout a temperature range from ambient temperature to 300 °C. According to TL theory, when the heating rate increases and the peak intensity steadily decreases, the glow peak's peak temperature changes towards higher temperatures. Thermal quenching, where quenching effectiveness increases at higher temperatures, is responsible for the observed drop in TL glow peak intensity with increasing heating rates. There is a significant 22% drop in peak intensity when maximal TL intensities are normalized to the lowest heating rate (2 °C/s).

Keywords: activation energy, nano α-alumina, thermoluminescence, heating rate.

1. Introduction

The heating rate has a significant impact on the thermoluminescence (TL) properties of various materials. The effect of heating rate on the glow curve of MgB_4O_7:Tm,Dy has also been studied by González et al. (2022), who found that the kinetics parameters can be accurately determined using the sequential quadratic programming glow curve deconvolution (SQPGCD) method. This is consistent with the findings of Cruz-Zaragoza et al. (2011), who also observed changes in peak temperatures, peak intensities, and total area of glow peaks with increasing heating rates in other materials. Cruz-Zaragoza et al. (2011) found that as the heating rate increases, the peak intensity at the maximum decreases and shifts to a higher temperature. This was also observed by Ogundare (2005) in the case of fluorite, where the glow-peak temperatures increased with heating rate. Piters (1999) highlighted the influence of a temperature lag on the TL analysis, which can lead to a decrease in activation energy and frequency factor. Kitis (1993) studied the effects of heating rate on the TL glow-peaks of different phosphors, providing a comprehensive comparison of experimental results and theoretical calculations. These studies collectively demonstrate the significant impact of heating rate on TL properties [1].

The thermoluminescence glow curve of alumina (Al_2O_3) is influenced by the heating rate, with the response of single-crystal detectors decaying exponentially and ceramic detectors decaying linearly. Thermoluminescence of α-Al_2O_3:C irradiated to low doses (0.04–7.20 mGy) similar to those measurable in the environment has been investigated. The glow curve consists of a dominant peak near 200 °C and two additional glow peaks of weaker intensity at around 74 and 342 °C, respectively, for measurements made at 5 °C/s. Analysis of the main peak using various methods shows that in this low dose range, the peak follows non-first order kinetics and also that the peak is unitary rather than being a composite of two overlapping peaks as is thought to be the case at comparatively higher doses [2]. Its dose response was observed to be linear, and the dependence of the luminescence intensity on heating rate showed that the thermoluminescence is subject to thermal quenching [3]. However, further examination suggests that thermal quenching may be more of an effect with an increase in luminescence excitation dose. However, Kumar (2006) cautions that the conservation of area under the glow curve is not always guaranteed and that the glow peak height can increase with heating rate [4].

Considering the preceding discussions, the objective of this study was to explore the thermoluminescence (TL) characteristics of alpha-alumina (α-Al_2O_3) when subjected to varying radiation doses and heating rates. The focus was on analyzing the dose-response relationship and conducting kinetic analysis of the primary glow peak [5].

2. Materials and methods

In this study, nano-sized α-Al_2O_3 particles with sizes of 40 nm commercially available from Skyspring Nanomaterials, Inc. were used as samples [6–10].

Samples were scanned in the range $5 \leq 2\Theta \leq 80°$ at a 1.2°/min scan rate. Semi-quantitative estimates of mineral phase abundances were derived from the PXRD data using the intensity of specific reflections, density, and mass absorption coefficients of the elements for CuKα radiation. The crystallite size was calculated using OriginPro according to the formula:

$$D = \frac{K\lambda}{\beta \, cos\theta},$$

where D = Crystallite size; β=line broadening at FWHM in radians; θ (Bragg angle) = peak center; λ = X-ray wavelength; K (Dimensionless shape factor) = 0.94 for spherical crystallites. The samples were irradiated at ambient temperature with a ^{60}Co gamma source with a dose rate of 1.76 Gy/s. The dose rate was determined using a Magnette Miniscope MS400 EPR spectrometer with individually packed BioMax alanine dosimetry films with barcode markings developed by Eastman Kodak Company. The Harshaw TLD 3500 Manual Reader was utilized to assess the TL sample characteristics using a linear heating rate of 2 °C/s from 323 K to 673 K in an N_2 atmosphere with a Pilkington HA-3 heat-absorbing filter [11–16]. Three aliquots of 5 mg each were used for each measurement, and the TL data points represented the average of the three aliquots. A thin layer of the sample powder was uniformly distributed on the planchet surface to ensure a uniform TL signal [17].

3. Results and discussion

Dose response

The low-dose responses of nano α-Al_2O_3 with a particle size of 40 nm were evaluated within the dose range of 0.2 kGy to 8 kGy, and TL glow curves were recorded (Figure 1). Figure 1 reveals a distinct and prominent peak in the TL glow curve, with its maximum occurring at approximately 202 ± 2 °C. Notably, this TL peak exhibits a tendency to shift towards lower temperatures with an increase in dose level, which is not within the bounds of experimental error [18].

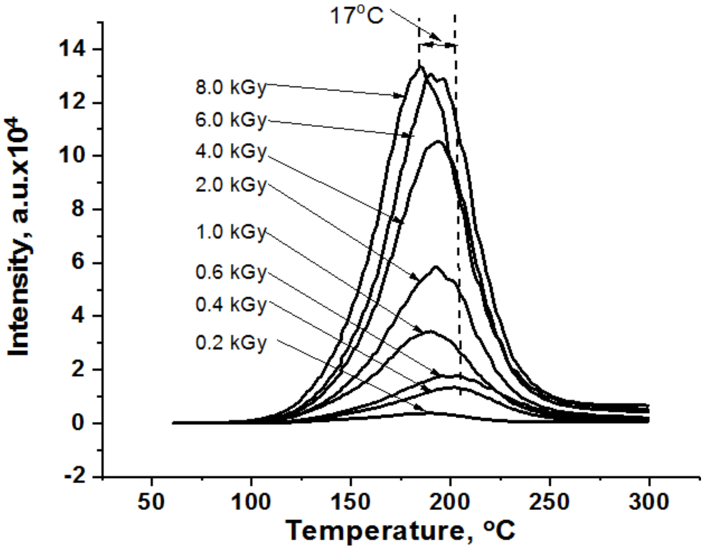
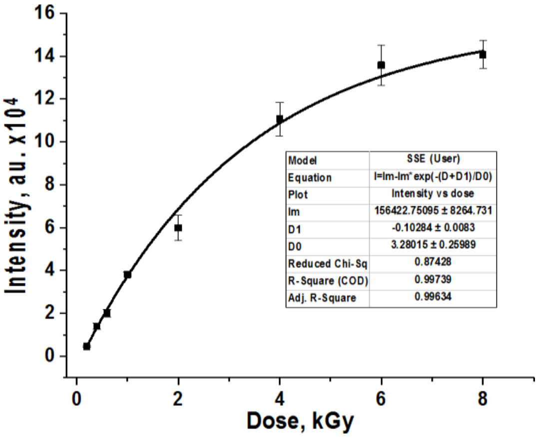


Fig. 1. TL glow curves of nano-α-Al_2O_3 obtained with a heating rate of 2 °C/s

The peak maximum temperature ($T_{\max}$) and the maximum intensity (I_{max}) are fundamental characteristics of a thermoluminescence (TL) peak. It is both theoretically predicted and experimentally verified that ($T_{\max}$) shifts to higher temperatures as the readout heating rate increases. Additionally, it is theoretically predicted that ($T_{\max}$) shifts to lower temperatures as the radiation dose increases. According to TL theory, peak temperatures are anticipated to change solely with the heating rate for first-order kinetics (order of kinetics b = 1). Consequently, under a constant heating rate, the peak maximum should remain relatively stable, unaffected by other experimental parameters and within the limits of experimental errors [19–20]. Therefore, if the TL peak temperature decreases with rising dose levels, it indicates non-first-order kinetics ($b \neq 1$), otherwise, it suggests first-order kinetics (b = 1). In this case the temperature shift is approximately 17 °C. The relationship between TL response and dose for nano α-Al_2O_3 with a particle size of 40 nm is depicted in Figure 2.



Model	SSE (User)
Equation	I=Im-Im*exp(-(D+D1)/D0)
Plot	Intensity vs dose
Im	156422.75095 ± 8264.731
D1	-0.10284 ± 0.0083
D0	3.28015 ± 0.25989
Reduced Chi-Sq	0.87428
R-Square (COD)	0.99739
Adj. R-Square	0.99634

Fig. 2. Dose dependence of TL intensity of nano-α-Al_2O_3 obtained at heating rate of 2 °C/s

The one-trap-one-recombination (OTOR) model is a basis for analyzing the effect of radiation dose on T_{max}, which predicts the behavior of the TL curve as well as the dose-response pattern. However, the empirical general-order kinetic equation helps to understand the nature of this effect. In this model, the dependence of T_{max} on radiation dose, which is mainly evident for second-order kinetics [21–25], gradually weakens with decreasing kinetic order and finally disappears for first-order kinetics. Despite the extensive focus on dose-response studies in the TL literature, the shift of T_{max} as a function of dose has not been clearly demonstrated experimentally.

Impact of Heating Rate

The influence of heating rate on TL glow curves is a fundamental experimental variable in TL measurements. The heating rate applied to dosimetric materials affects the variation in their TL sensitivity and, consequently, the trends observed in the dose curve.

In TL dosimetry applications, alterations in heating rate impact the TL glow peak (or curve) area and TL glow peak height. To assess the effect of heating rate on the TL glow curve and calculate kinetic parameters for nano α-Al_2O_3 with a particle size of 40 nm, samples were irradiated with a 6 kGy dose, and TL glow curves were recorded from room temperature to 300°C using various heating rates (2, 4, 6, 8, and 12 °C/s).

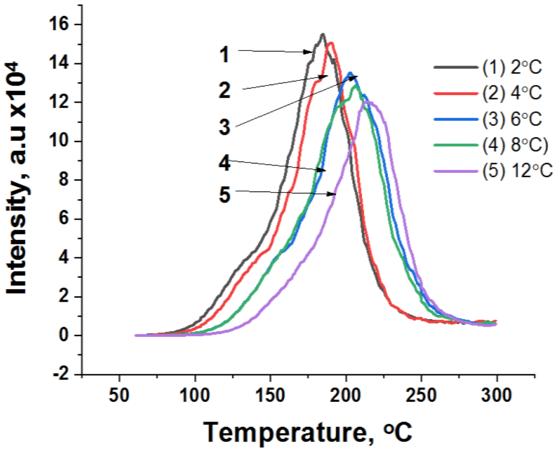


Fig. 3. TL glow curve of nano-α-Al_2O_3 at different heating rates

Figure 3 displays TL glow curves of the irradiated samples, indicating that the peak temperature of the glow peak shifts towards higher temperatures as the heating rate increases and the peak intensity continuously diminishes, aligning with TL theory.

The maximum value of the main dosimetry peak temperature is close to that determined for alumina doped with carbon, with a predominant TL peak centered at 471 K and α-Al_2O_3, where $T_m = 465$ K, but significantly higher than that determined for alumina crystal ($T_m = 450$ K), bauxite ($T_m = 412$ K), and α-Al_2O_3 doped with Sr, Li, and Ge ($T_m = 448$ K). Natural diaspores exhibit TL glow curves with a low-temperature maximum peaked at 453 K and a wide broad curve above 490 K. Diapores samples show a discrete distribution of electron traps at a lower temperature (~463 K) and a continuous structure of traps at a higher temperature (above 500 K), which is due to dehydroxylation and oxidation of the chromophore. Al_2O_3 nanoparticles doped with Cr (particle size of 25 nm) show a prominent peak at approximately 474 K and a linear response from 100 Gy to 20 kGy.

4. Conclusions

The basic TL and XRD characteristics of irradiated nano-α-alumina particles were investigated in this work, especially how they reacted to different heating rates. The observed TL glow curves showed that the peak continuously moved toward lower temperatures as the radiation exposure increased. This behavior reveals additional information about the kinetic processes of nano-α-alumina by showing that the TL peak follows non-first-order kinetics.

Heating rates emerged as a critical experimental variable, significantly influencing the dosimetric performance of nano α-Al_2O_3. The TL glow peak temperature shifted to higher values as the heating rate increased, while peak intensity diminished. This behavior aligns with established TL theory, offering valuable information for optimizing the use of nano α-Al_2O_3 in dosimetric applications under different heating conditions.

The decrease in TL glow peak intensity with higher heating rates was attributed to thermal quenching, where quenching efficiency rises with temperature. This finding highlights the importance of considering thermal quenching effects in the development and refinement of TL materials.

Notably, the study revealed an exponential increase in TL intensity with increasing radiation dose for the exposed nanoparticles. This suggests that alumina nanoparticle powder could serve as a promising substrate material for ionizing radiation dosimetry, opening up new possibilities for its application in radiation monitoring and dosimetric technologies. Further development could focus on enhancing sensitivity and exploring its use across a broader range of radiation environments.

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ТЕМПЕРАТУРНО-ЗАВИСИМЫЕ ВНУТРЕННИЕ ПАРАМЕТРЫ ТЕРМОЛЮМИНЕСЦЕНЦИИ НАНО-АЛЮМИНИЯ

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Резюме: В данной работе исследуются основные свойства термолюминесценции облучённых наночастиц α-оксида алюминия, а также их поведение при различных скоростях нагрева. Для анализа регистрировались кривые термолюминесценции (TL), которые демонстрируют чёткий пик с максимумом примерно при 202°C. При увеличении дозы пик постепенно смещается в сторону более низких температур, что свидетельствует о кинетике, отличной от первого порядка ($b \neq 1$). С помощью рентгенодифракционного анализа (XRD) был определён размер кристаллитов, который составил около 40 нм. Образцы были облучены дозой 6 кГр для определения кинетических параметров нано-α-Al_2O_3 и изучения влияния скорости нагрева на кривую свечения. Кривые термолюминесценции регистрировались при различных скоростях нагрева (2, 4, 6, 8 и 12 °C/с) в диапазоне температур от комнатной до 300 °C. Согласно теории термолюминесценции, при увеличении скорости нагрева температура максимума пика смещается в сторону более высоких температур, тогда как интенсивность пика постепенно уменьшается. Наблюдаемое снижение интенсивности пика связано с термическим тушением, эффективность которого возрастает при более высоких температурах. Отмечено значительное снижение интенсивности пика на 22% при нормировании максимальных значений TL по отношению к наименьшей скорости нагрева (2 °C/с).

Ключевые слова: энергия активации, нано-α-оксид алюминия, термолюминесценция, скорость нагрева.

NANO-ALÜMİNİUMUN TERMOLUMİNESENSİYASININ TEMPERATURDAN ASILI DAXİLİ PARAMETRLƏRİ

Ə.B. Əhədov, S.Q. Məmmədov, A.S. Əhədova

Xülasə: Bu tədqiqatda şüalanmış nano-α-alüminium oksid hissəciklərinin əsas termolüminesensiya xüsusiyyətləri və onların müxtəlif qızdırılma sürətlərinə reaksiyası araşdırılmışdır. Tədqiqat üçün TL (termolüminesensiya) əyriləri qeydə alınmış və təxminən 202 °C temperaturda maksimuma malik aydın pik müşahidə edilmişdir. Doza səviyyəsi artdıqca pik daha aşağı temperaturlara doğru sürüşür ki, bu da birinci tərtib olmayan kinetikaya ($b \neq 1$) uyğun gəlir. XRD analizi vasitəsilə kristallit ölçüsü müəyyən edilmiş və onun təxminən 40 nm olduğu aşkar edilmişdir. Nümunələr nano α-Al_2O_3 üçün kinetik parametrləri müəyyən etmək və qızdırılma sürətinin TL işıqlanma əyrisinə təsirini öyrənmək məqsədilə 6 kGy doza ilə şüalandırılmışdır. Daha sonra TL əyriləri müxtəlif qızdırılma sürətlərində (2, 4, 6, 8 və 12 °C/s) otaq temperaturundan 300 °C-yə qədər intervalda qeydə alınmışdır. Termolüminesensiya nəzəriyyəsinə görə, qızdırılma sürəti artdıqca pik temperaturu daha yüksək temperaturlara doğru dəyişir, eyni zamanda pik intensivliyi tədricən azalır. Qızdırılma sürətinin artması ilə TL pik intensivliyində müşahidə olunan azalma termal söndürülmə ilə əlaqədardır və bu effekt yüksək temperaturlarda daha güclü olur. Minimum qızdırılma sürəti (2 °C/s) ilə müqayisədə maksimal TL intensivliklərinin normallaşdırılması nəticəsində pik intensivliyində 22% azalma qeydə alınmışdır.

Açar sözlər: aktivləşmə enerjisi, nano α-alüminium oksidi, termolüminesensiya, qızdırılma sürəti.