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# THE EFFECT OF HEATING RATE OF NANO-A-ALUMINA ON THERMOLUMINESCENCE

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This research explores the fundamental thermoluminescence characteristics of irradiated nano- $\alpha$ -alumina particles, investigating their response to varying heating rates. The study involves recording TL luminescence curves, revealing a distinct peak with a maximum at approximately 202°C. As dose levels increase, the peak consistently shifts towards lower temperatures, indicating adherence to non-first-order kinetics (b≠1). To examine the impact of the heating rate on the TL glow curve and derive kinetic parameters for nano  $\alpha$ -Al2O3 with a particle size of 40 nm, specimens were exposed to a 6 kGy dose. Subsequently, TL glow curves were documented over a temperature range from room temperature to 300°C, employing different heating rates (2, 4, 6, 8, and 12°C/s). 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 observed decrease in TL glow peak intensity with escalating heating rates is attributed to thermal quenching, where quenching efficiency rises at higher temperatures. Normalizing maximum TL intensities to the lowest heating rate ( $2^{\circ}C/s$ ) reveals a substantial 22% decrease in peak intensity.

*Keywords:* Activation energy; Nano  $\alpha$ -alumina; Thermoluminescence; heating rate

## **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 MgB4O7:Tm,Dy has also been studied by [1], 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 [2], who also observed changes in peak temperatures, peak intensities, and total area of glow peaks with increasing heating rates in other materials. Cruz-Zaragoza [2] 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 [3] in the case of fluorite, where the glow-peak temperatures increased with heating rate. Piters [4] highlighted the influence of a temperature lag on the TL analysis, which can lead to a decrease in activation energy and frequency factor. Kitis [5] 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.

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 [6]. Thermoluminescence of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>: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 dose. 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. Howe-11 JOURNAL OF YOUNG RESEARCHER, 2024, Nº 4, ISSN 2409-4838

ver, further examination suggests that thermal quenching may be more of an effect with increase in luminescence excitation dose [7]. However, Kumar [8] 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.

Considering the preceding discussions, the objective of this study was to explore the thermoluminescence (TL) characteristics of alpha-alumina ( $\alpha$ -Al<sub>2</sub>O<sub>3</sub>) 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.

## MATERIAL AND METHODS

In this study, nano-sized  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> particles with sizes of 40 nm commercially available from Skyspring Nanomaterials, Inc. were used as samples. The samples were irradiated at ambient temperature with a <sup>60</sup>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 323K to 673K in an N<sub>2</sub> atmosphere with a Pilkington HA-3 heat-absorbing filter. 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.

#### **RESULTS AND DISCUSSION**

#### 1. Dose response and dose equation

The low-dose responses of nano  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> 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 (refer to Figure 1). Figure 1 reveals a distinct and prominent peak in the TL glow curve, with its maximum occurring at approximately 202±2°C.



Fig. 1. TL glow curves of nano- $\alpha$ -Al<sub>2</sub>O<sub>3</sub> obtained with heating rate of 2°C/s

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.

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 [9]. Therefore, if the TL peak temperature decreases with rising dose levels, indicating non-first-order kinetics (b≠1), otherwise, it suggests first-order kinetics (b=1). In this case temperature shift is approximately  $17^{\circ}$ C. The relationship between TL response and dose for nano  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> with a particle size of 40 nm is depicted in Figure 2.



Fig. 2. Dose dependence of TL intensity of nano- $\alpha$ -Al<sub>2</sub>O<sub>3</sub> obtained at heating rate of 2°C/s

# 2. Impact of Heating Rate



Fig. 3. TL glow curve of nano- $\alpha$ -Al<sub>2</sub>O<sub>3</sub> at different heating rates

The influence of heating rate on TL glow curves is a fundamental experimental variable in TL measurements [10]. The heating rate applied to dosimetric materials affects the variation in their TL sensitivity and, consequently, the trends observed in the dose curve [3]. In TL dosimetry applications, alterations in heating rate impact the TL glow peak (or curve) area and TL glow peak height [11]. To assess the effect of heating rate on the TL glow curve and calculate kinetic parameters for nano  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> 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). 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 [12].



**Fig. 4.** Normalized TL glow curve intensity at different heating rates

Numerous studies have reported a decrease in TL glow peak height (or area) intensity with an increase in heating rate. This phenomenon is attributed to thermal quenching, whose efficiency escalates with rising temperatures [3],[13], [14]. Normalizing the maximum TL intensities of the glow peaks to the lowest heating rate (2°C/s) reveals a roughly 22% decrease in peak intensity (Figure 4).

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 [15] and  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, where T<sub>m</sub>=465 K [16], but significantly higher than that determined for alumina crystal (T<sub>m</sub>=450 K) [5], bauxite (T<sub>m</sub>=412 K) [17], and Al<sub>2</sub>O<sub>3</sub> doped with Sr, Li, and Ge (T<sub>m</sub>=448 K) [18]. Natural diaspores exhibit TL glow curves with a low-temperature maximum peaked at 453 K and a wide broad curve above 490 K [19]. 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 [20]. Al<sub>2</sub>O<sub>3</sub> 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 kG [21].

#### CONCLUSION

This study delved into the fundamental thermoluminescence characteristics of irradiated nano- $\alpha$ -alumina particles, studying how they respond to different heating rates. During our studies, TL luminescence curves were recorded, which revealed a distinct peak reaching a maximum at approximately 202°C. Notably, as the dose levels increased, the peak exhibited a consistent shift towards lower temperatures. This intriguing observation led us to conclude that the thermoluminescent peak adheres to non-first-order kinetics (b $\neq$ 1).

The influence of heating rates emerged as a pivotal experimental variable in our study. Dosimetric materials, particularly nano  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, demonstrated shifts in TL sensitivity and variations in dose curve trends in response to different heating rates. As the heating rate increased, the TL glow peak temperature shifted towards higher values, accompanied by a diminishing peak intensity. This aligns seamlessly with the expectations of TL theory.

The decrease in TL glow peak intensity with escalating heating rates was elucidated as a consequence of thermal quenching, where the efficiency of quenching rises with higher temperatures. A significant revelation emerged when we normalized the maximum TL intensities to the lowest heating rate (2°C/s), unveiling a substantial 22% decrease in peak intensity[22], [23], [24], [25], [26]. Adding to the significance of our exploration, we observed a exponential increase in TL intensity of the exposed nanoparticles with the radiation dose. This points towards the potential use of alumina nanoparticle powder as a substrate material for ionizing radiation dosimetry, opening up promising avenues for practical applications in this field.

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# NANO-ALFA-ALÜMİNİUM OKSİDİNİN QIZDIRMA SÜRƏTİNİN TERMOLÜMİNİSENSIYAYA TƏSİRİ

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

Bu tədqiqat şüalanmış nano-α-alüminium hissəciklərinin əsas termolüminesans xüsusiyyətlərini araşdırır, onların müxtəlif istilik dərəcələrinə reaksiyasını araşdırır. Tədqiqat TL-nin lüminesans əyrilərinin qeydə alınmasını əhatə edir, maksimum təxminən 202°C-də fərqli bir zirvə aşkar edir. Doza səviyyələri artdıqca, pik ardıcıl olaraq daha aşağı temperaturlara doğru dəyişir və birinci dərəcəli olmayan kinetikaya (b≠1) riayət olunmasını göstərir.

Qızdırma sürətinin TL parıltı əyrisinə təsirini araşdırmaq və hissəcik ölçüsü 40 nm olan nano α-Al2O3 üçün kinetik parametrləri əldə etmək üçün nümunələr 6 kGy dozaya məruz qalmışdır. Sonradan, TL parıltı əyriləri müxtəlif istilik dərəcələrindən (2, 4, 6, 8 və 12°C/s) istifadə etməklə otaq temperaturundan 300°C-yə qədər olan temperatur intervalında sənədləşdirilmişdir. Parıltı pikinin pik temperaturu qızdırma sürəti artdıqca daha yüksək temperaturlara doğru dəyişir və pik intensivliyi TL nəzəriyyəsinə uyğun olaraq davamlı olaraq azalır.

Artan istilik dərəcələri ilə TL parıltısının pik intensivliyində müşahidə edilən azalma, daha yüksək temperaturlarda söndürmə səmərəliliyinin yüksəldiyi termal söndürmə ilə əlaqələndirilir. Maksimum TL intensivliklərinin ən aşağı qızdırma sürətinə (2°C/s) normallaşdırılması pik intensivliyin əhəmiyyətli dərəcədə 22% azaldığını aşkar edir.

**Açar sözlər:** Aktivləşdirmə enerjisi; Nano α-alüminium oksidi; termolüminesans; istilik dərəcəsi

# ВЛИЯНИЕ СКОРОСТИ НАГРЕВА НАНО-А-ОКСИДА АЛЮМИНИЯ НА ТЕРМОЛЮМИНЕСЦЕНЦИЮ

# А.Б. Ахадов, С.Г. Мамедов, А.Ш. Ахадова

В этом исследовании изучаются фундаментальные термолюминесцентные характеристики облученных частиц нано-α-оксида алюминия, исследуется их реакция на различные скорости нагрева. Исследование включает регистрацию кривых люминесценции ТЛ, выявляющих отчетливый пик с максимумом приблизительно при 202°С. По мере увеличения уровней дозы пик последовательно смещается в сторону более низких температур, что указывает на соблюдение кинетики не первого порядка (b≠1).

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# FİZİKA-RİYAZİYYAT VƏ TEXNİKA ELMLƏRİ

Чтобы изучить влияние скорости нагрева на кривую свечения ТЛ и вывести кинетические параметры для нано- $\alpha$ -Al<sub>2</sub>O<sub>3</sub> с размером частиц 40 нм, образцы подвергались воздействию дозы 6 кГр. Впоследствии кривые свечения ТЛ были задокументированы в диапазоне температур от комнатной температуры до 300 °C, используя различные скорости нагрева (2, 4, 6, 8 и 12 °C/c). Пиковая температура пика свечения смещается в сторону более высоких температур по мере увеличения скорости нагрева, а интенсивность пика непрерывно уменьшается, что соответствует теории ТЛ. Наблюдаемое снижение интенсивности пика свечения TL с ростом скорости нагрева объясняется термическим гашением, где эффективность гашения возрастает при более высоких температурах. Нормализация максимальных интенсивности ТL к самой низкой скорости нагрева (2°C/c) показывает существенное снижение интенсивности пика на 22%.

**Ключевые слова:** Энергия активации; Нано а-оксид алюминия; Термолюминесценция; скорость нагрева