DOSIMETRIC PROPERTIES OF ALUMINIUM OXIDE DOPED WITH GERMANIUM CO-DOPED WITH STRONTIUM IRRADIATED BY COBALT-60 GAMMA RAYS

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ABSTRACT

The thermoluminescence (TL) properties of powder Al_2O_3, Al_2O_3:Ge and Al_2O_3:Ge;Sr irradiated by Cobalt-60 gamma rays were studied. The TL properties investigated were the minimum detectable dose, glow curve, trap parameters, linearity, sensitivity, thermal fading and reproducibility. Samples were prepared using combustion synthesis technique with various heat treatments to study the influence on the TL response. The physical properties of the samples were determined using X-Ray diffraction (XRD), energy dispersive X-ray field emission scanning electron microscopy (EDX-FESEM) and transmission electron microscopy (TEM). The experiments showed that Al_2O_3:Ge (3.0%);Sr (0.3%) was the best in terms of TL properties compared to other prepared samples. The annealing procedure of the sample was determined at 400 °C for 1h, followed by 80 °C for 16 h with the heating rate of 3 ºC s^{-1}. Analysis of kinetic parameters showed that the sample possessed second order kinetic characteristics supported by the whole glow peak technique. Its activation energy, obtained by peak shape method was $E = 1.37 \pm 0.14$ eV and its frequency factor, $s = 4.70 \pm 1.18 \text{ x } 10^{17}\ s^{-1}$, however by using initial rise method, $E = 1.00 \pm 0.01$ eV and $s = 4.36 \pm 1.67 \times 10^{10}\ s^{-1}$. The samples exhibited good sensitivity at high dose (10 – 80 Gy) and low dose (0.5 – 4.0 Gy) with 52.72 nC $mg^{-1}\ Gy^{-1}$ and 43.40 nC $mg^{-1}\ Gy^{-1}$ respectively. In addition, the linearity of the sample was also good at high dose and low dose with correlation coefficient, R^2 of 0.9681 and 0.9961 respectively. The fading was low at high dose (50 Gy) and low dose (4 Gy) with the percentage loss of 33% and 34% after 60 days exposed. Furthermore, it also possessed good reproducibility with the percentage of standard deviation of 2.4% (4 Gy) and 6.5% (50 Gy) with the minimum detectable dose of 0.0065 mGy. The existence of main peaks in XRD pattern, TEM micrographs and the results of EDX-FESEM clarified that the sample was in the nano polycrystalline state with size of 9.36 nm and all the required elements were present in the powder sample. In conclusion, Ge 3.0% mol doped, Sr 0.3 mol% co-doped Al_2O_3, was found to be a potential material to be used as a TL dosimeter which satisfies most of the desirable TL characteristics.

ABSTRAK

Ciri-ciri termopendarcahaya (TL) serbuk Al_2O_3, Al_2O_3:Ge dan Al_2O_3:Ge;Sr disinari oleh sinar gama Co-60 telah dikaji. Sifat-sifat termopendarcahaya yang telah dikaji ialah dos pengesanan minimum, lengkung berbara, parameter perangkap, kelinearan, kepekaan, kelunturan terma dan kebolehgunaan semula. Sampel-sampel ini telah disediakan menggunakan teknik sintesis pembakaran dengan pelbagai rawatan haba untuk mengkaji pengaruhnya terhadap tindakbalas termopendarcahaya. Sifat-sifat fizikal sampel ditentukan dengan menggunakan belauan sinar-X (XRD), serakan tenaga sinar-X pancaran medan pengimbas elektron mikroskop (EDX-FESEM) dan mikroskopi elektron penghantaran (TEM). Keputusan eksperimen menunjukkan Al_2O_3:Ge(3.0%);Sr(0.3%) mempunyai sifat TL yang lebih baik berbanding sampel lain yang disediakan. Prosedur penyepuhlindapan sampel ini telah ditentukan pada 400 °C selama 1 jam, diikuti 80 °C selama 16 jam, dengan kadar pemanasan 3 °C s^{-1}. Analisis parameter kinetik menunjukkan sampel mempunyai sifat kinetik tertib kedua dan disokong oleh teknik puncak berbara keseluruhan. Nilai tenaga pengaktifan sampel diperolehi dengan kaedah bentuk puncak ialah $E = 1.37 \pm 0.14$ eV dan faktor frekuensi, ialah $s = 4.70 \pm 1.18 \times 10^{17}$ s^{-1}, namun begitu dengan menggunakan kaedah kenaikan awal $E = 1.00 \pm 0.01$ eV dan $s = 4.36 \pm 1.67 \times 10^{10}$ s^{-1}. Sampel ini mempamerkan kepekaan yang baik pada julat dos tinggi (10 Gy – 80 Gy) dan julat dos rendah (0.5 Gy – 4.0 Gy) masing-masing dengan 52.72 nC mg^{-1} Gy^{-1} dan 43.40 nC mg^{-1} Gy^{-1}. Tambahan pula, kelinearan sampel baik pada julat dos tinggi dan julat dos rendah masing-masing dengan pekali korelasi, R^2 iaitu 0.9681 dan 0.9961. Sampel ini juga menunjukkan kepudaran yang rendah pada dos yang tinggi (50 Gy) dan dos yang rendah (4 Gy) dengan peratusan isyarat yang hilang adalah 33% dan 34% selepas 60 hari didedahkan. Tambahan pula, ia juga mempunyai kebolehgunaan semula yang baik dengan peratusan sisihan piawai 2.4% (4 Gy) and 6.5% (50 Gy) dengan dos pengesanan minimum ialah 0.0065 mGy. Kewujudan beberapa puncak utama pada corak XRD, mikrograf TEM dan keputusan EDX-FESEM membuktikan sampel tersebut adalah dalam keadaan nano polihablur dengan saiz 9.36 nm dan terdapat semua unsur yang diperlukan dalam sampel serbuk. Kesimpulannya, Al_2O_3 didop Ge 3.0 mol%, dikodop Sr 0.3 mol% didapati merupakan bahan yang berpotensi untuk digunakan sebagai dosimeter TL yang memenuhi ciri-ciri TL yang diingini

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LIST OF ABBREVIATIONS

ALARA	-	As Low As Reasonably Achievable
CB	-	Conduction band
EDX	-	Energy dispersive X-ray
ERD	-	Environmental radiation dosimetry
FESEM	-	Field emotion scan electron microscopy
ICRP	-	International commission on radiological protection
ICRU	-	International commission of radiation unite
KE	-	Kinetic energy
LINAC	-	Linear accelerator
LLP	-	Lowest level of detection
PMT	-	Photomultiplier Tube
PRD	-	Personal radiation dosimetry
RD	-	Radiation dosimetry
RF	-	Radio frequency
TD	-	Threshold dose
TL	-	Thermoluminescence
TLD	-	Thermoluminescence dosimetry
TMAC	-	Total mass attenuation coefficient
TTP	-	Time temperature profile
UKM	-	Universiti Kebangsaan Malaysia
UPMU	-	University Laboratory Managemnet Unit
VB	-	Valence band
WinREMS	-	Window Radiation Evaluation and Management System
XRD	-	X-Ray diffraction

LIST OF SYMBOLS

σ_B	-	Standard deviation
$\overline{B}$	-	Mean TL background signal from non-irradiated samples
$(RER)_E$	-	Relative Energy Response
μ_g	-	Geometrical factor
^{60}Co	-	Cobalt-60
A	-	Total activity of the radio nuclide
Ai	-	Atomic mass
$Al_2 (NO_3)_3.9H_2O$	-	Aluminium Nitrate
Al_2O_3	-	Aluminium Oxide
Ao	-	Initial activity of the radionuclide
Ar	-	Area under the glow curve
b	-	Order of kinetics
$CO(NH_2)_2$	-	Urea
D	-	Absorbed Dose
$D (t)$	-	Radiation dose at time
D_0	-	Minimum detectable dose
E	-	Activation energy
Ef	-	Energy level
E_g	-	Energy band gap
F	-	Calibration factor of TL system
F	-	Field Strength
$f (D)$	-	Linearity index
F(D)	-	Dose response at dose D
$F(D_1)$	-	Dose response at linear dose D_1
GeO_2	-	Germanium oxide
I	-	Intercept-m
I	-	TL intensity
Im	-	Maximum TL intensity
k	-	Boltzman constant
Li_2NO_3	-	Lithium Nitrate

m	-	Mass
m	-	Concentration of recombination centers
m	-	Slope from linearity graph
Mi	-	average molecular weight
M_T	-	Total molecular weight
n	-	Refractive index
n	-	Number of trapped electron
N	-	Trap concentration
N_A	-	Avogadro's number
n_o	-	Number of trapped electrons at t_o=0
R	-	Recombination center
R^2	-	Regression coefficient
s	-	Frequency factor
S	-	Sensitivity
$S_E (E)$	-	Photon energy response
$Sr (NO)_3$	-	Strontium Nitrate
S_d	-	Relative sensitivity
t	-	Time
T	-	Temperature
$t_{1/2}$	-	Half-life
T_1	-	The lower temperature of the *Tm* corresponding to the half peak intensity
T_2	-	The higher temperature of the *Tm*corresponding to the half peak intensity
Tm	-	Peak temperature
V_M	-	Molar volume
W_i	-	Weight fraction of element
x	-	Mole fraction
Z	-	Atomic number
Z_{eff}	-	Effective atomic number
β	-	Heating rate
δ	-	High temperature half width
ρ	-	density

σ_B	-	Standard deviation from mean background signal
τ	-	Mean lifetime
ω	-	Total half intensity width

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Ionizing radiation is a radiation that has enough energy to ionize atoms and molecules when it interacts with matter. It can be detected and measured using various instruments. However, ionizing radiation has the potential to damage living tissue depending on the type or amount of ionizing radiation that is absorbed by that particular tissue. [1]. Hence, radiation detection is important for protecting people and the environment from harmful radiations [2].

Dosimeter is a tool which is commonly used to identify the quantity of absorbed dose in a body. This device is a key tool in monitoring environmental, personal, and medical treatment dose. Thermoluminescence dosimeter (TLD) is one of the most popular personal dosimeter used to estimate the radiation dose deposited in an individual [3]. The importance of thermoluminescence for radiation dosimetry is due to the fact that the amount of light emitted is propotional to the absorbed dose by the irradiated material, which require sensitive detection and accurate measurement of ionizing radiation [4].The use of the TL technique in the field of radiation dosimetry has started by Daniels [5] after finding the benefit of lithium fluoride (LiF) in the present decade. TL material should be able to retain trapped electrons in some periods of time at a suitable temperature with a suitable environment and have strong light output. The stored energy will be released in form of visible light when the material is heated. The energy stored in the crystal is released with the emission of light as a function of temperature forming a glow curve while the irradiated material was heated. The position, shape and intensities of the glow peaks are important properties for TL [6].

Various preparation techniques and characteristics of numerous TL materials has been studied until today. There are many criteria that must be full filled before the sample is declared as TL materials. The materials also, should have high sensitivity and linearity, low fading, good stability through multiple of reading sample and its effective atomic mass (Z_{eff})is close to human tissue [7]. The latter is very important because materials with higher or lower Z_{eff} than human tissue could influence the contributions of high energy photons leading to a radiation damage. Furthermore, based on previous research [8]–[10], the materials should have high accuracy, precision and resistivity with environmental factors such as humidity, light, gasses, and moisture and should be strong, chemically inert and radiation resistant.

However, not all TL substances are appropriate to expect to possess the above mentioned properties. Hence, it is essential to find the best preparation procedures and the properties of the materials. Recently, the investigation of TL materials has been explored and extended to the optical fibre and nanoparticle [11]. Distinct preparation procedure and characteristics of many TL materials have been studied until now to improve the TLD performance [12].

During 1957, the dosimetric properties of aluminium oxide (Al_2O_3) were found by Rieke [13][14]. But this material was forgotten for a long time because there was no enhancement on sensitivity performance compared to TLD 100 [15]. However, the research on these materials suddenly increased after the development of Al_2O_3 in crystals form [16]. Alpha alumina or known as corundum has been on attraction as one of most important ceramic material due to its significant properties such as high melting point, thermal conductivity, hardness and high strength at elevated temperature. To approach alpha phase, high temperature annealing above 1100ºC of production is mandatory [17]–[19].

As an example, α-Al_2O_3:C crystal, which was first introduced by Akselrod [20] is now being used extensively as an element for TLD making. As aluminium oxide displays an effective number almost near to that of human tissue (Z_{eff} = 11.28), therefore, it is considered as a better choice to use in environmental and medical dosimetry [21]. Grown α - Al_2O_3:C crystal demonstrates excellent properties of TL as it holds a better TL sensitivity towards α, β or γ radiations. It shows a sensitivity of approximately 40 - 60 times higher than TLD-100 at a heating rate of 4 °Cs^{-1}, one glow peak at 460 K as well as low background including low noticeable threshold limit i. e. 0.1 *μGy* with N_2 flow, lower fading throughout storage (in dark) and a linear dose-response of maximum 10 Gy [22] .

The unique characteristics of Al_2O_3:C demonstrates a high sensitivity towards ionizing radiation with a low detectable dose, extended luminescence lifetime of recombination making attainable that rapid pulsed- optically stimulated luminescence (OSL) measurement process and gradual decay permitting multiple absorbed radiation dose determinations. Due to this characteristics, the sensitivity of these detectors varies strongly with the rate of heating of the material, which is an undesirable source of errors for it use [23].From the previous studies in synthesizing α - Al_2O_3 powder, it involves solid state transformation from the hydrates of aluminium oxide. The process occurs on heating around 1230 °C [24]. Besides that, other conventional fabrication techniques of those dosimeters follow Vernuil or Czochralsky crystal growth methods, which also includes a high temperature (>2000°C) [25]. Hence, this research explores a different preparation method to overcome high-temperature dependent process. In previous research, combustion synthesis (CS) technique has come out as an excellent method to make crystalline materials due to its low temperature of processing.

A sustained effort has been made towards the improvements of the sensitivity of Al_2O_3. For example, previous research [26]–[28] used Si, Ti, Mg, Cr and Yi as a dopant. However, this dopant still did not improve the sensitivity of the TL performance. Until now, the decision on the right choice of the dopants are still uncertain and it is believed that the dopants play the major role in TL phenomenon.Inspired by these notable attributes of Al_2O_3, this study intends to introduce new and completely different type of dopant which is Germanium (Ge) and Strontium (Sr) nanoparticle powder in dosimeter material to enhanced the measurement of dosage. In addition, the TL properties of aluminium oxide, doped with Ge and co-doped with Sr are thoroughly examined. This three samples are expected to enhance the TL intensity and can improve its performance. All results are analyzed, interpreted and discussed to draw a comparison on each other. This newly proposed TL material is expected to be a potential used in TLD in the future.

1.2 Problem Statement

The commercially available dosimeter based alumina named TLD 500 is in the form of single crystal and has sensitivity of 40 to 60 times greater than TLD-100 in ionizing radiation [29]. Since 1950, a lot of research has been performed on the thermoluminescence of alumina (Al_2O_3) with different dopants [30]. Doped aluminium oxide (α-Al_2O_3) has been extensively used as a luminescent dosimeter for radiation. This material was first introduced because of its high sensitivity and grown in a highly reducing atmosphere hence, creates a large concentration of oxygen vacancies [31].

Conventionally, this material is prepared by crystals growth method, which entails sophisticated laboratory equipment, a very high temperature (approx.2050°C) processing including a high reducing atmosphere [32][25]. In order to reduce the processing temperature, CS method is considered to be a precise technique to make crystalline materials due to its minimal cost, lower processing temperature, rapid reaction time and the capability to attain high purity single phase oxide powders [33].

Recent studies reveal that the nanomaterials produced by the CS method influence luminescence of certain materials on thermoluminescence study[34]. A special attention has also been made on the improvement of TL response upon heat treatment. Thus, due to such preliminary remarkable output observed in these nanomaterials, attention is given in nanosized phosphors owing to few positive points results in detection of high energy ionizing radiations [1]. These benefits include of providing fundamental knowledge of newly doped aluminium oxide powder sample that may lead to many other novel TL dosimetry applications.

1.3 Objectives

The objectives of this study are:

a) To determine the optimum concentration of Ge doped and Sr co-doped Al_2O_3 by using CS method.

b) To compare the influence of heating rate and the effect of heat treatment towards TL intensity response.

c) To evaluate the thermoluminescence properties of the samples in terms of glow curve, sensitivity, linearity, fading, reproducibility, minimum detectable dose limit and TL kinetic parameters.

d) To characterize the samples in terms of crystallinity nature, its elemental composition and stability by using Energy Dispersive Analysis X-Ray (EDAX), Field Emission Scanning Electron Microscopy (FESEM) and Transmission Electron Microscopy (TEM).

1.4 Scope of the Study

A new Al_2O_3 powder doped with germanium and strontium act as dopants and co-dopants are synthesized via combustion synthesis to evaluate their TL performance. The best optimum concentration of doped materials was compared followed by the co-doped sample after being exposed to Cobalt-60 gamma irradiation. The TL properties such as linearity, sensitivity, glow curve, fading and reproducibility were also investigated. The kinetic analysis of the glow curve was done to obtain the activation energy and frequency factor. These analyses were done in order to get a better understanding on TL phenomenon.

The optimum sample from TL response was characterized via X-Ray diffraction (XRD); in addition, elemental distribution was detected by Energy

Dispersive X-Ray (EDX) analysis. EDX was done to confirm the presence of impurities. Prepared samples were calcined at a different temperature from 500°C to 1400°C for 2 hours to study the effect of heat treatment towards TL response.

In order to accurately determine the particle size and trace whether the sample are in the nanocrystalline phase, transmission electron microscopy (TEM) was done. All samples were irradiated with Cobalt-60 gamma rays ranged from 0.5- 4.0Gy and 10 Gy to 80 Gy at Universiti Kebangsaan Malaysia. All samples were read out utilizing TLD reader model Harshaw 3500 at UTM Nuclear Laboratory.

1.5 Significance of the Study

The present study on Ge-doped and Sr co-doped Al_2O_3 may exploit the new knowledge and additional information for new TL material that can be used in TLD system besides improve the intensity of TL performance. The current study is expected to promote better understanding on new material properties with optimum composition for accurate dose measurement leading to human safety. Based on this research, a new Al_2O_3 doped with Ge as dopant and co doped with Sr are synthesized via combustion synthesis method. The present study on doped and co-doped Al_2O_3 nanoparticle prepared by combustion offer valuable information and is expected to provide good enhancement of TL performance through discover of a new improved TL material. In addition, the performance of these TLD materials can be used in TLD systems for clinical, personal and environmental monitoring purposes.

1.6 Thesis Outline

Chapter 1 briefed an introduction on basic studies of thermoluminescence including the research background, problem statement, objectives, scope and significance of this research. Chapter 2 reveals more about the literature review for CS methods, thermoluminescence theory and the presentation of previous studies of aluminium oxide and others elements as TLD. Chapter 3 describes the methodology to prepare nanoparticles doped and co-doped Al_2O_3 powder form based on CS method to fulfil the proposed objective. This chapter also encloses the instrumentation and analysis used for sample characterization for this work. Next, Chapter 4 explained the experimental data attained from this research. The results describe the optimization of dopants and co-dopants, temperature time profile, effect of heating rate towards thermoluminescence response, details the main dosimetric properties of the proposed TLD with structure and morphology. Lastly, Chapter 5 concludes the thesis outcome with some limitation of this research and recommendation for future outlook.

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LIST OF PUBLICATIONS

1. **Saharin,N.S.,**Ahmad,N.E.,Tajuddin,H.A.,Tamuri,A.R., Thermoluminescence properties of aluminium oxide doped strontium, lithium and germanium prepared by combustion synthesis method. *EPJ Web Conf.* 2017, 156, 1–8.

2. **Saharin, N.S..,** Ahmad, N.E., Tamuri, A.R., Thermoluminescence study of aluminium oxide doped germanium prepared by combustion synthesis method. *EPJ Web Conf.* 2017, 156, 1–7.

3. Tajuddin, H.A., Wanhassan, W.M.S., Abdul Sani, S.F., **Shaharin, N.S.,** Thermoluminescent properties of dy doped calcium borate based glass for dose measurement subjected to photon irradiation. *EPJ Web Conf.* 2017, 156.