THERMOLUMINESCENCE DOSIMETRIC PROPERTIES OF GERMANIUM DOPED, GERMANIUM-BORON DOPED OPTICAL FIBRES AND CALCIUM BORATE DOPED DYSPROSIUM NANOMATERIAL

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DEDICATION

I dedicate this work

To my dear parents and parents in law

Whose love, kindness, patience and their prayer have brought me this far

To my husband

Mohd Hairi Bin Mohd Shafie

For his love, understanding and support through my endeavour

To my lovely children

Arissa Amani

Damia Darwisyah

Naifal Nuqman

Hadif Harraz

Whose presence fills my life with joy

To my siblings

For their endless laughs and tears

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ABSTRACT

Thermoluminescent dosimetric properties of germanium doped and germanium-boron doped optical fibres, and dysprosium doped calcium borate nanomaterial were determined. The optical fibres were produced via modified chemical vapour deposition while dysprosium doped calcium borate nanomaterial was prepared via solution combustion synthesis. The objective was to develop new thermoluminescent material for radiation dose measurements of high energy electron beam. The interest in high dose measurements is due to numerous applications of electron beam in radiation technologies such as health care product sterilization, polymer modification and food processing. Characterization of samples was made by X-ray diffraction (XRD), energy dispersive X-ray (EDX) and transmission electron microscope (TEM) analyses. The particle size of dysprosium doped calcium borate (CaB_4O_7:Dy) nanomaterial influenced the thermoluminescent signal. From TEM analysis, the average crystal size of CaB_4O_7:Dy was found to be 12.5 nm. A well-defined peak appeared with maximum thermoluminescent response around 180 °C to 200 °C. Both germanium doped and germanium-boron doped optical fibres showed good linearity for dose range of 1 kGy – 150 kGy with correlation coefficient R^2 of 0.931 and 0.957, respectively. CaB_4O_7:Dy nanomaterial also showed good linearity response with $R^2 = 0.825$ and its response saturated beyond 100 kGy. Effective atomic number Z_{eff} were 13.5 for CaB_4O_7:Dy, 14.4 for germanium doped optical fibre and 15.3 for germanium-boron doped optical fibres, respectively. Germanium doped optical fibres showed the best results for reproducibility and fading with the values of 7.4% and 4% after 60 days of storage. Germanium-boron doped optical fibres showed the highest sensitivity (82.34 $nC.mg^{-1}.Gy^{-1}$) compared to germanium doped optical fibre (70.68 $nC.mg^{-1}.Gy^{-1}$) and CaB_4O_7:Dy nanomaterial (23.02 $nC.mg^{-1}.Gy^{-1}$). The trap parameters namely kinetic order k, activation energy E and frequency factor s were also determined. The values were, $E = 0.722$ eV, $s = 2.33 \times 10^8 s^{-1}$; $E = 0.547$ eV, $s = 7.63 \times 10^6 s^{-1}$; and $E = 0.515$ eV, $s = 3.11 \times 10^6 s^{-1}$ for germanium-boron doped, germanium doped optical fibres and CaB_4O_7:Dy nanomaterial, respectively. From the values of geometric factors (Balarin's formula), germanium-boron doped optical fibre and CaB_4O_7:Dy obeyed the second kinetics order while germanium doped optical fibre obeyed first kinetics order. This work indicates potential use of germanium doped and germanium-boron doped optical fibres as dosimeters in high energy electron beam irradiation applications.

ABSTRAK

Sifat-sifat dosimetri termopendarcahaya bagi gentian optik didop germanium dan didop germanium-boron, dan nanobahan kalsium borat didop disprosium telah ditentukan. Gentian-gentian optik dibuat khas melalui kaedah pemendapan wap terubahsuai dan nanobahan kalsium borat didop disprosium telah dihasilkan melalui sintesis pembakaran larutan. Objektif utama kajian adalah untuk menghasilkan bahan pengesan termopendarcahaya baharu untuk pengukuran dos sinaran alur elektron tenaga tinggi. Minat dalam pengukuran dos tinggi adalah disebabkan oleh pelbagai aplikasi alur elektron dalam teknologi sinaran seperti pensterilan produk penjagaan kesihatan, pengubahsuaian polimer dan pemprosesan makanan. Pencirian sampel dibuat dengan menggunakan pembelauan sinar-X (XRD), serakan tenaga sinar-X (EDX) dan analisis mikroskop elektron penghantaran (TEM). Saiz zarah nanobahan kalsium borat didop dysprosium (CaB_4O_7:Dy) mempengaruhi isyarat termopendarcahaya. Daripada analisis TEM, saiz purata hablur CaB_4O_7:Dy ialah 12.5 nm. Puncak yang jelas dengan sambutan termopendarcahaya maksimum muncul di sekitar 180 ℃ hingga 200 ℃. Kedua-dua gentian optik yang didop germanium dan germanium-boron menunjukkan sambutan kelinearan yang baik untuk julat dos 1 kGy – 150 kGy dengan pekali korelasi R^2 masing-masing bernilai 0.931 dan 0.957. Nanobahan CaB_4O_7:Dy juga menunjukkan sambutan kelinearan yang baik dengan R^2 = 0.825 dan sambutannya tepu apabila melebihi 100 kGy. Nombor atom berkesan Z_{eff} masing-masing ialah 13.5 untuk CaB_4O_7:Dy, 14.4 untuk gentian optik didop germanium dan 15.3 untuk gentian optik didop germanium-boron. Gentian optik didop germanium-boron menunjukkan kepekaan paling tinggi (82.34 $nC.mg^{-1}.Gy^{-1}$) berbanding dengan gentian optik didop germanium (70.68 $nC.mg^{-1}.Gy^{-1}$) dan nanobahan CaB_4O_7:Dy (23.02 $nC.mg^{-1}.Gy^{-1}$). Parameter-parameter perangkap iaitu tertib kinetik k, tenaga pengaktifan E dan faktor frekuensi s juga ditentukan. Nilai-nilainya ialah E = 0.722 eV, $s = 2.33 \times 10^8$ s^{-1}; E = 0.547 eV, $s = 7.63 \times 10^6$ s^{-1}; dan E = 0.515 eV, $s = 3.11 \times 10^6$ s^{-1} masing-masing bagi gentian optik didop germanium-boron, gentian optik didop germanium dan CaB_4O_7:Dy. Daripada nilai faktor geometri (rumus Balarin), gentian optik didop germanium-boron dan CaB_4O_7:Dy mematuhi tertib kinetik kedua manakala gentian optik didop germanium mematuhi tertib kinetik pertama. Hasil kajian ini menunjukkan potensi penggunaan gentian optik didop germanium dan germanium-boron sebagai dosimeter dalam aplikasi penyinaran alur elektron tenaga tinggi.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

BG	-	Band Gap
ECC	-	Element Correction Coefficient
EBM	-	Electron beam machine
EDAXS	-	Energy Dispersive X-ray Spectroscopy
FESEM	-	Field Emission Scanning Electron Microscope
Ge	-	Germanium
Ge-B	-	Germanium-Boron
Gy	-	Gray
ICRP	-	International Commission on Radiological Protection
ICRU	-	International Commission of Radiation Units
IR	-	Initial rise
kGy	-	Kilo Gray
MCVD	-	Modified Chemical Vapour Deposition
MMU	-	Multimedia University
MNA	-	Malaysia Nuclear Agency
NPs	-	Nanoparticles
PCF	-	Photonic-crystal fibre
PMT	-	Photomultiplier Tube
R^2	-	Correlation Coefficient
RCF	-	Reader Correction Factor
SCS	-	Solution combustion synthesis
STD	-	Standard Deviation
$T_{1/2}$	-	Half-life
TEM	-	Transmission Electron Microscope
TL	-	Thermoluminescence
TLD	-	Thermoluminescence dosimetry
TTP	-	Time/Temperature Profile
UKM	-	Universiti Kebangsaan Malaysia
UV	-	Ultraviolet
XRD	-	X-ray Diffraction

LIST OF SYMBOLS

Au	-	Gold
B_2O_3	-	Boron Trioxide
CaB_4O_7	-	Calcium tetraborate
$CaCO_3$	-	Calcium Carbonate
CaF	-	Calcium Fluoride
CaO	-	Calcium Oxide
cm	-	Centimetre
Co-60	-	Cobalt-60
Cu	-	Copper
Dy_2O_3	-	Dysprosium
Eu	-	Europium
Ge_2	-	Germanium
$GeCl_4$	-	Germanium Tetrachloride
Gy	-	Gray
H_3BO_3	-	Boric acid
LiF	-	Lithium Fluorite
Li_2O	-	Lithium Oxide
mA	-	Mili-Ampere
MeV	-	Mega electron volt
MgO	-	Magnesium Oxide
Mn	-	Manganese
nC	-	Nano-coulomb
O_2	-	Oxygen
Pb	-	Lead
$SiCl_4$	-	Silica Tetrachloride
SiO_2	-	Silica Dioxide
TLD-100	-	Lithium Fluorite doped with Magnesium and Titanium
a	-	Fractional contribution
A_w	-	Atomic mass

$\mathring{A}$	-	Angstrom
b	-	Order of kinetics
$^{\circ}C$	-	Degree Celsius
D	-	Average crystalline size
E	-	Activation energy
k	-	Boltzmann Constant
N_e	-	Number of electrons per gram
N_A	-	Avogadro's number
P	-	Trap
R	-	Heating rate
S	-	Sensitivity, Stopping power
s	-	Frequency factor
T	-	Temperature
t	-	Time
$T_{1/2}$	-	Half life
T_M	-	Maximum temperature
Wi	-	Fractional weight
W	-	Fraction of the Element
Z	-	Atomic number
Z_{eff}	-	Effective atomic number
$3D$	-	Three Dimensional
λ	-	Wavelength
γ	-	Gamma Rays
v	-	Frequency
θ	-	theta
μm	-	Micro-meter
g	-	Gram
β	-	Diffracted full width
S	-	Stopping Power
ρ	-	density
Ic	-	Glow intensity
I_M	-	Maximum Intensity
μ_g	-	Geometric factor

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Overview

Thermoluminescence dosimetry (TLD) is a study of materials which emit light when heated after exposure to ionizing radiation. It has been a mainstay of health physics for over 50 years. During that time, TLDs have been extremely valuable in monitoring the safety of radiation workers and performing environmental dose control. The use of TLD as a method for radiation dosimetry of ionizing radiation has been extended for many technologies applications including radiation modification of the properties for various materials, medical product sterilization, and disinfection of agricultural product, food irradiation industry and nuclear reactor monitoring (Kozlov et al., 2006). The major advantages of TL dosimeters are their small physical size, high sensitivity, reusable with only a gradual change in efficiency and economical (Bhatt, 2011). Unfortunately, not all types of thermoluminescent dosimeters can meet all the requirements. Some are best used at low energy range while others at high energy range. Their sensitivity could also vary thus the problem of selecting TLD depends on the task they are used for. These served as the reasons for more extensive study for design and development of high dose TL dosimeter materials.

There are two types of TL measurement device, active (e.g. ionizing chambers) and passive (e.g. luminescence dosimeters). The TL method has been the focus of current research, seeking knowledge in fabrication or synthesizing new thermoluminescence dosimetry (TLD) media for radiation dosimetry, working towards the media in which amount of light emitted is proportional to the dose absorbed by the irradiated material. Indeed, novel TLD materials based on doped optical fibres which currently being study, offer TL characteristics that provide good potential for extending their application (Issa et al., 2011; Bradley et al., 2012). Commercially available optical fibres made for telecommunications purposes (with outer diameter typically ~100 μm) have been seen to offer the basis of highly sensitive spatial resolution dosimeters, such as measuring doses in medical radiations applications. Studies of the dosimetric properties of commercially produced Ge-doped SiO_2 optical fibres have been performed by several research groups using x-ray microbeam radiation as well as for kilovoltage to high energy (megavoltage) photon and electron beams, demonstrating the potential of Ge-doped SiO_2 optical fibres as a novel form of TL dosimeter (Rahman et al., 2010 ; Hashim et al., 2010; Issa et al., 2011).

Several commercial TL dosimeters providing high sensitivity and low fading, however cannot provide accurate estimation of doses for high range of exposure that even need to be measured in technological applications (Tekin et al., 2010). The studies do not focus restrictively on the fabricating of new materials but also include investigations of the defects present within the structure of the material. By adding suitable dopants to the base material optical fibres (SiO_2) for instance, has demonstrated an ability to enhance the TL yield of the material. A study by Benabdesselam et al. (2013) showed that a Ge-doped optical fibre is very attractive for TL dosimetry application.

The studies in nanomaterial ceramics as new TLD material are showing results that point to these materials as good candidate media especially in high dose measurements. Recently, interest of researchers towards nanomaterials has increased because of the enhanced optical, electronic and structural properties of these materials. Pandey et al. (2002) and Pandey et al. (2003) reported some TL characteristics of a couple of nanomaterials. Salah et al. (2007) have synthesized nanocrystalline of some highly sensitive phosphors like $CaSO_4$: Dy, LiF: Mg, Cu, P, MgB_4O_7: Dy, $K_2Ca_2(SO_4)_3$: Eu and K_3NaSO_4: Eu and studied their TL response to different ionizing radiations (Salah et al., 2006; Salah et al., 2007; Lochab et al., 2007). They found very interesting results, especially the response of these nanomaterials to very high dose. Due to these good results obtained in nanomaterials, it is of great importance to further study the TL properties of different nanomaterials to heavy doses of ionizing radiations. Calcium tetraborate CaB_4O_7 and calcium metaborate CaB_2O_4 polycrystalline ceramics are some of the most attractive systems among the borate compounds as they present good chemical stability due to their very low hygroscopic nature (Santiago et al., 2001).

1.2 Problem Statement

According to Chen and McKeever (1997), high dose is a term typically referring to absorb doses in the range 1000 Gy – 10^6 Gy. There are many applications for such irradiations including sterilization of medical equipment products, disinfection of agricultural products and radiation control of defects in large machine parts. Other than that, polymer products such as cable jackets, car tyres, pipes and packaging materials as well as food products are the most common media benefitting from high dose irradiation services.

Thermoluminescence (TL) dosimetry at high dose radiation has attracted much attention as increasing needs in food safety, radiation protection on extreme dose field and nuclear reactor dose monitoring. Therefore, it is crucial to investigate the performance of a dosimeter under circumstances of high dose irradiation. Besides that, the TL mechanism including the TL response and glow curve behaviour especially in the supralinearity need to be studied. Currently, about twenty types of commercial TL detectors with 10 μGy – 20 Gy dose measurement range are widely used for personal dosimetry and radiation monitoring of environment (Kortov, 2007). Still, the choice of TL detectors for high-dose dosimetry is limited. Two types of TL detectors are used in USA; CaF_2: Mn (TLD-400) with operating dose range 10^{-6} Gy – 10^2 Gy and $Li_2B_4O_7$: Mn (TLD-800) with the dose range 10^{-3} Gy – 10^5 Gy. In Russia, TL detector on the base of alumina phosphate (PST) and sodium silicate (TLD-K) glasses with the highest registered dose of 2 kGy – 5 kGy are widely used (Aluker et al., 2006). A great number of storage materials are known to be promising for development of high dose TLD. However, there are challenges such as saturation of the TL yield at high dose irradiation. Therefore, this study will measure TL yield in wide range of electron beam irradiation doses (1 kGy up to 150 kGy).

For the past few years, researchers have realized the potential of optical fibres as new TL material due to the smaller diameter size (~ 100 μm) for sensitive in-vivo radiotherapy assessment at high spatial resolution (Hashim et al., 2009). There are findings showing that commercial doped optical fibres can provide ~ 10 times greater response to radiation than commercial phosphor based TLD-500 (Benabdesselam et al., 2013). Concerning advances, doped optical fibres are relatively new material in TL dosimeters but have attracted considerable attention and application-compatible properties, including use in challenging environments such as potentially damp/limpid or even aqueous environments. Most previous studies concerning the TL doped optical fibres have tendency to focus on medical dosimetry applications such as radiotherapy with maximum doses of Gy. In addition, the doped optical fibres offer good reproducibility and reusability without loss to dose response (Espinosa et al., 2006). The purpose of present study is to evaluate the TL characteristics of potential doped optical fibres for high dose dosimetry application.

CaB_4O_7: Dy nanomaterial is a relatively unstudied TL phosphor which is suggested as an alternative to LiF (TLD-100) for the dosimetry of ionizing radiation because of its linear response dose, negligible fading and excellent reusability (Dhoble et al., 2004; Proki´c, 2000). According to Kortov (2014), nanostructured phosphors are the most suitable materials for high dose detectors. They have big capacity of trapping canters due to the surface of the centres. The nanostructured materials show unique features that cannot obtain from the conventional macroscopic materials. The surface states are very important to the physical properties, especially the optical properties of the nanoparticles (Sharma et al., 2011). As the particle size decrease to nano-scale, the surface to volume ratio and consequently the numbers of surface states increase. Those significant properties of nanomaterials have been motivated further studies of various nanomaterials for high doses measurements. Therefore, one of the objectives of this research was to study the TL properties of new potential CaB_4O_7: Dy nanomaterials as TL materials for high dose radiation.

Although the use of the doped optical fibres is clearly great interest, the process of producing the optical fibres via the Modified Chemical Vapour Deposition (MCVD) route is a complicated procedure. This process requires a range of high technology facilities and the use of higher temperatures, from 1000 °C up to 2000 °C. Conversely, the solution combustion synthesis (SCS) offers a much simple processing technique of producing nanomaterial samples for TL purpose (Rivera & Azorin, 2007; Shafiqah et al., 2015; Wang et al., 2012). Additionally, an alternative method based on the solution combustion technique involving a wet chemistry process, concerns producing surface decorated or doped process to the nanoparticles samples.

In this study, the samples are fabricated by using two different methods. The custom-made optical fibres were produced via MCVD method and CaB_4O_7: Dy nanomaterial was prepared by using the solution combustion technique. This research investigates the performance of Ge-doped and Ge-B-doped optical fibres and calcium tetraborate doped with dysprosium (Dy) nanomaterial. In general, this study investigates these potential materials as TL dosimeters in terms of their preparation, structural characteristics and thermoluminescence properties.

1.3 Objectives of Study

i. To synthesize dysprosium (Dy) doped calcium borate nanoparticle by using solution combustion synthesis (SCS) method.
ii. To utilize the tailor-made Ge-doped and Ge-B-doped optical fibres (fabricated via MCVD) for high dose irradiation.
iii. To investigate the structural properties, elemental composition and morphology (i.e., Energy Dispersive X-Ray, X-ray Diffraction and Transmission Electron Microscope) of nanophosphors material.
iv. To evaluate the thermoluminescence (TL) dosimetric properties of the synthesized samples irradiated to electron beam accelerator (high dose).
v. To compare the dosimetric properties of Dy doped calcium borate with tailor-made optical fibres and TLD-100.

1.4 Research Question

In order to throw some lights on the TL dosimetry problems, it would be better to look into the basic principle of thermoluminescene. To narrow down the scope of searching the knowledge in this area, the following questions are used as guidance:

1. What is the suitable method to be used for producing the TL materials?
2. What are the experimental procedures to be used in order to investigate the optimum TL materials?
3. What are the TL characteristics of the tailor-made optical fibres and nanomaterial sample?

1.5 Scope of Study

The main focus in this work is to evaluate TL characteristics of different types of potential TL materials as options for high dose dosimetry application. In this study, the ionizing radiation that commonly used for commercial high dose industry such as electron beam and Co-60 (γ-rays) have been used throughout this research. The samples were irradiated to wide dose range from 1 kGy up to 150 kGy by using electron beam accelerator at Malaysia Nuclear Agency. Other than TL characteristics study, the structural properties for the synthesized samples such as X-ray diffraction (XRD), Energy dispersive X-ray (EDX) and transmission electron microscope (TEM) were also studied.

1.6 Significance of Study

Regardless the wide availability of different thermoluminescence material for dosimetry, there is continued interest in the development of new dosimetric materials in the dosimetry of high-LET radiation and the dosimeters which can be used in high dose industry. Some basic limitations of the luminescence dosimeters must be considered in seeking new materials. The optical fibres Ge-doped and Ge-B-doped have opened the way to use this material for measuring the dose in the high and the ultra-high range, particularly for regular monitoring of ionizing radiation around the high-energy accelerators and nuclear reactors facilities during emergency cases. Thus this study was aimed to evaluate the TL dosimetric properties of Ge-B-doped and Ge-doped optical fibres and CaB_4O_7: Dy nanomaterial toward high dose irradiation.

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