UNIVERSITI PUTRA MALAYSIA

NANOSYNTHESIS, AND STRUCTURAL AND DOSIMETRIC CHARACTERISTICS OF UNDOPED AND COPPER MANGANESE-DOPED CALCIUM BORATE THERMOLUMINESCENT DOSIMETERS

MARYAM ERFANI HAGHIRI

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By

MARYAM ERFANI HAGHIRI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

July 2013

DEDICATION

In appreciation of their love, sacrifices, faith, and eternal goodness

I would like to dedicate my thesis to my dear mother

and

To the memory of my father, my grandmother and my beloved fiancé

Ebrahim

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

NANOSYNTHESIS, AND STRUCTURAL AND DOSIMETRIC CHARACTERISTICS OF UNDOPED AND MANGANESE COPPER-DOPED CALCIUM BORATE THERMOLUMINESCENT DOSIMETERS

By

MARYAM ERFANI HAGHIRI

July 2013

Chairman: Professor Elias Saion, PhD

Faculty: Science

Tissue equivalent thermoluminescent dosimeters (TLDs) are an effective device to measure low and high absorbed doses of ionizing radiation in protected area, medical and industrial applications or as a personal monitoring dosimeter. A number of commercially available TLDs are common for this purpose where the TL intensity is proportional to absorbed dose but they are of a narrow dose range. In this research efforts were made to enhance the present TL performance of these materials to a wider dose range by employment of nanosynthesis method and introducing impurities to the TL materials. The un-doped and Cu-Mn doped calcium tetraborate nanocrystals (CaB_4O_7) were fabricated using the combination of co-precipitation and heat-treatment methods. For the un-doped samples, 100 ml de-ionized water containing 0.2-mol calcium chloride ($CaCl_2$), 0.5-3 wt% of Polyvinyl pyrrolidone (PVP) were mixed with 100 ml de-ionized water containing 0.2-mol borax

($Na_2B_4O_7$) drop wise to form fine white precipitations of calcium tetraborate (CaB_4O_7). For the doped samples, manganese chloride ($MnCl_2$) and copper chloride ($CuCl_2$) were added into the PVP solution before mixing with borax. The precipitations were then centrifuged (3500 rpm for 10 min) and washed several times with distill water before drying at 80 ^oC for 24 h and annealing at temperatures between 700 to 970 ^oC and at fixed annealing times of 1 to 5 h. The synthesized un-doped and Cu-Mn doped CaB_4O_7 nanocrystals were used to investigate the structural and thermoluminescent characteristics.

The XRD patterns of synthesized nanparticles at initial precipitation showed almost crystalline structure as compared to those undergone thermal treatment at the annealing temperatures of 750 to 900 ^oC where the dominant phase structure became ortothrombic structure. Increasing annealing temperature from 700 to 970 ^oC, the aggregation of the nanoparticles enlarged and the average particle size increased from 5.5 to 14 nm as measured by the transmission electron microscopy (TEM). The extension of annealing time did not show a significant change in the conversion of phase structure except small variation in the peak positions. The mono-dispersed and spherical shape calcium borate nanoparticles were realized using 1-wt% PVP stabilizer to produce the average particle sizes of 5.5 nm at the initial precipitation and 8 nm at the optimum annealing temperature of 970 ^oC for 1 hour annealing time.

The manganese doped calcium borate (CaB_4O_7:Mn) nanocrystals showed a single TL peak at around 149 ^oC with enhanced TL sensitivity over the un-doped CaB_4O_7 nanocrystals by 60 times and produced a good linearity response from 0.05 to 2000

Gy. However, it showed high degree of fading of 52 % per month and failed to become a good TLD phosphor. The copper doped calcium borate (CaB_4O_7:Cu) nanocrystals showed two prominent TL peaks located at 114 and 246 ^oC. The TL sensitivity of low and high temperature peaks of (CaB_4O_7:Cu) nanocrystals increased by 1 and 3 times compared to un-doped samples, respectively. The remarkable feature of this nanophosphor is on its wide range linear dose response from 0.05 to 3000 Gy for the high temperature peak of 246 ^oC and its limited range linear dose response from 0.05 to 30 Gy for the low temperature peak of 114 ^oC. The fading was 26 % in 2 months storaged in a dark room for the high temperature peak and a poor fading for the low temperature peak after 1 week.

The TL response of double doped calcium borate (CaBT:Cu-Mn) nanocrystal showed two prominent TL peaks located at 124 and 256 ^oC. The incorporation of manganese into the host lattice, as a co-dopant, increased the TL efficiency of low and high temperature peaks by 2.30 and 3.67 times more than un-doped samples, respectively. The double doped nanophosphor revealed an excellent linearity dose respond in the range of 0.05 to 3000 Gy for both temperature peaks. The low temperature peak displayed a considerable fading after 1 week storage, whilst the high temperature peak presented the fading of 23 % after 2 month storage in a dark room.

As a comparative study, the TL sensitivity of single and double doped calcium borate nanocrystals were compared with the one of the standard and commercial TL dosimeter, TLD 100 (LiF:Mg,Ti) after exposure to 10 Gy gamma dose. The results

demonstrated that the manganese (1.4 mol%) doped calcium borate nanocrystal has a sensitivity of 2.75 times higher than the TLD100. However, the sensitivity of copper doped and copper-manganese CaBT nanocrystals at this particular exposure (i.e. 10 Gy) is less than that of TLD-100 phosphor by a factor of approximately 0.04 and 0.09 times for low temperature peak and 0.13 and 0.14 for high temperature peak, respectively.

Abstrak tesis yang dikemukakan kepada senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

SINTISIS NANO, DAN CIRI-CIRI STRUKTUR DAN DOSIMETRI DOSIMETER TERMOLUMINASEN KALSIUM BORATE TANPA DOPAN DAN BERDOPAN-KUPRUM MANGANAM

Oleh

MARYAM ERFANI

Julai 2013

Pengerusi: Profesor Elias Saion, PhD

Fakulti: Sains

Tisu setara dosimeter thermoluminescent (TLD) adalah suatu alat yang berkesan untuk mengukur dos terserap sinaran mengion paras rendah dan tinggi dalam kawasan dilindungi, aplikasi dalam perubatan dan industri atau sebagai dosimeter pemantauan peribadi. Beberapa TLD komersial boleh didapati yang lazimnya digunakan untuk tujuan ini di mana keamatan TL adalah berkadar dengan dos terserap tetapi mereka mempunyai sela dos terhad. Dalam kajian ini, usaha telah dibuat untuk meningkatkan prestasi bahan TL sedia ada kepada sela dos yang lebih luas dengan mengunakan kaedah sintisis nano dan menambahkan bahan asing dalam bahan TL. Hablur nano kalsium tetraborate (CaB_4O_7) tanpa dopan dan berdopan Cu-Mn telah direkabentuk dengan menggunakan kesatuan kaedah pemendakan dan kaedah rawatan terma. Bagi sampel tanpa dopan, 100 ml air nyah ion mengandungi

0.2 mol kalsium klorida ($CaCl_2$), 0.5-3% berat polivinil pyrrolidone (PVP) telah ditambahkan kedalam 100 ml air nyah ion yang mengandungi 0.2 mol borak ($Na_2B_4O_7$) secara pentitisan untuk membentuk mendak putih kalsium tetraborak (CaB_4O_7). Bagi sampel berdopan, manganam klorida ($MnCl_2$) dan kuprum klorida ($CuCl_2$) telah ditambah ke dalam larutan PVP sebelum ditambahkan dengan borak. Mendapan itu kemudian kitar (3500 pusingan per minit selama 10 minit) dan dibasuh dengan air suling beberapa kali sebelum dikeringkan pada suhu 80 ^oC selama 24 jam dan penyepuhlindapan pada suhu 700-970 ^oC dan pada masa penyepuhlindapan daripada 1 hingga 5 jam. Hablur nano CaB_4O_7 yang disediakan tanpa dopan dan berdopan Cu-Mn telah digunakan untuk menyiasat ciri-ciri struktur dan thermoluminesen.

Corak XRD zarah nano yang disediakan pada peringkat awal mendakan menunjukkan struktur hampir hablur berbanding dengan yang menjalani rawatan terma pada suhu penyepuhlindapan 750-900 ^oC yang mana struktur fasa menjadi struktur ortothrombic. Dengan meningkatkan suhu penyepuhlindapan 700-970 ^oC, pengagregatan nanopartikel membesar dan purata saiz zarah meningkat 5.5-14 nm seperti yang diukur menggunakan mikroskop elektron transmisi (TEM). Lanjutan masa penyepuhlindapan tidak menunjukkan perubahan yang ketara dalam pertukaran struktur fasa kecuali perubahan kecil terhadap kedudukan puncak. Kalsium borak bersifat pelaraian mono dan zarah nano berbentuk sfera telah dihasilkan dengan menggunakan 1% berat penstabil PVP untuk mendapatkan purata saiz zarah 5.5 nm pada mendapan awal dan 8 nm pada suhu penyepuhlindapan optimum 970 ^oC selama 1 jam masa penyepuhlindapan.

Hablur nano kalsium borak berdopan manganum (CaB_4O_7:Mn) menunjukkan puncak tunggal TL pada sekitar 149 ^oC dengan sensitiviti TL dipertingkatkan melebihi hablur nano kalsium borak tanpa dopan sebanyak 60 kali dan menghasilkan dos liner 05-2000 Gy. Walau bagaimanapun, ia mempunyai darjah kepudaran yang tinggi sebanyak 52% sebulan dan gagal untuk menjadi fosfor TLD yang baik. Hablur nano kalsium borak berdopan kuprum (CaB_4O_7:Cu) menunjukkan dua puncak TL yang menonjol terletak pada 114 dan 246 ^oC. Keamatan puncak TL suhu rendah dan tinggi (CaB_4O_7:Cu) meningkat masing-masing 1 dan 3 kali berbanding sampel tanpa dopan. Ciri-ciri fosfor nano yang luar biasa ini mempunyai pelbagai sambutan dos linear 05-3000 Gy untuk puncak suhu tinggi pada 246 ^oC dan sambutan dos linear dos terhad 05-30 Gy untuk puncak suhu rendah pada 114 ^oC. Bagaimanapun darah kepudaran adalah 26% untuk puncak suhu tinggi untuk tempoh 2 bulan penyimpanan di dalam bilik gelap dan darah kepudaran untuk puncak suhu rendah adalah hanya 1 minggu penyimpanan.

Sambutan TL hablur nano kalsium borak berdopan ganda (CaBT:Cu-Mn) menunjukkan dua puncak TL yang menonjol terletak di 124 dan 256 ^oC. Penambahan manganum ke dalam kekisi tuan rumah, sebagai dopan bersama, meningkat kecekapan puncak TL suhu rendah dan tinggi masing-masing sebanyak 2.30 dan 3.67 kali lebih baik daripada sampel tanpa dopan. Fosfor nano pendopan berganda menonjolkkan sambutan dos linear cemerlang dalam julat 05-3000 Gy untuk kedua-dua puncak suhu TL. Puncak suhu rendah bagaimanapun agak pudar

selepas penyimpanan 1 minggu, manakala puncak suhu tinggi darjah kepudaran sebanyak 23% selepas 2 bulan simpanan dalam bilik gelap.

Sebagai kajian perbandingan, sensitiviti TL kalsium yang didopkan secara tunggal dan berpasangan bersama borate nanokristal dibandingkan dengan salah satu alat pengukur dos TL yang berpiawai dan komersial, iaitu TLD 100 (LIF: Mg, Ti) selepas pendedahan kepada dos gamma 10 Gy. Keputusan menunjukkan bahawa mangan (1.4 mol%) yang didopkan bersama kalsium borate nanokristal mempunyai kepekaan 2.75 kali lebih tinggi daripada TLD100. Walaubagaimanapun, kepekaan tembaga yang didopkan dan tembaga-mangan nanokristal CaBT pada pendedahan dos tertentu (iaitu 10 Gy) adalah kurang daripada TLD-100 fosfor dengan faktor kira-kira 0.04 dan 0.09 kali untuk puncak yang bersuhu rendah dan 0.13 dan 0.14 kali untuk puncak yang bersuhu tinggi masing-masing.

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I also owe my thanks to my brothers, my nephews and all my family for their encouragement and love from thousands of miles away. I owe you all more than just my thanks. May God’s graces and blessings be forever yours.

I certify that a Thesis Examination Committee has met on July 2013 to conduct the final examination of Maryam Erfani Haghiri on her thesis entitled "**Nanosynthesis, structural and dosimetric characteristics of un-doped and manganese-copper doped calcium borate thermoluminescent dosimeters**" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The committee recommends that the student be awarded the Doctor of Philosophy.

Members of the Thesis Examination Committee were as follows:

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This thesis submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirements for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

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MARYAM ERFANI HAGHIRI

Date: 11 July 2013

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LIST OF ABBRIVIATIONS

^{60}Co	Cobalt-60
AR	Analytical Grade
CaBT	Calcium borate
CB	Conduction Band
CPE	Charged Particles Equilibrium
Cu	Copper
Dy	Dysprosium
EthoH	Ethanol
Eu	Europium
FTIR	Fourier Transforms Inferred
FWHM	Full Width Half Maximum
GCD	Glow Curve Deconvolution
GOT	General One Trap
Gy	Gray
ICCD	International Centre for Diffraction Data
IRCP	International Commission of Radiological Protection
k	Boltzmann ´s constant
ML	Multi Level model
Mn	Manganese
OSLD	Optically Stimulated Luminescence Dosimeter
PMT	Photomultiplier
PVP	Polyvinylpyrrolidone
Q.E	Quasi Equilibrium
R	Recombination center
SEM	Scanning Electron Microscope
SI	International Standard of Units
Sm	Samarium
SSD	Radiation Source to Sample Distance
TEM	Transmission Electron Microscope
TGA	Thermal Gravimetric Analysis
TIM	Track Interaction Model
TL	Thermoluminescence
TLD	Thermoluminescence Dosimeter
TSC	Thermally Stimulated Conductivity
TSL	Thermo Stimulated luminescence
VB	Valence Band
wt	Weight
XRD	X-ray Diffraction

YAG	Yttrium aluminium grant
Z_{eff}	Effective Atomic number

CHAPTER I

INTRODUCTION

1.1 Background of Study

Thermally Luminescence (TL) or more specifically Thermally Stimulated Luminescence (TSL) is the phenomenon of emission of lights from a solid either a semiconductor or an insulator which has been previously exposed to ionizing radiation under conditions of increasing temperature (Mandavia, 2011). Several studies on improving TL properties were carried out and a number of commercially thermoluminescent dosimeters (TLDs) are available for this purpose (Salah et al., 2009b). It is known that the phosphor materials have a limited dose - range that depends on their sensitivity to ionizing radiation. The difficulties of accurate estimation of dose response at high doses is due to saturation of the TL signal while at low doses, very low signal to noise makes it complex to assess the amount of dose (Salah et al., 2007). Due to growing interest in TLDs for the environmental, personal and clinical applications, extensive investigations have been focused on producing new and high performance TLD materials for a wide range of doses (Kitis et al., 2002). Currently, TL materials with nanometer dimensions such as quantum dots, nanowires, nanorods and nanotubes, have attracted a great deal of attention in different fields especially in the field of radiation monitoring (Sharma et al., 2009a). It has been found that the physical properties of individual nanoparticles can be quite different from the conventional macroscopic materials. Recent studies on different nanocrystalline materials have revealed the striking features such as high sensitivity

and high linear dose response whereas the conventional microcrystalline phosphors are not capable of (Salah et al., 2009b).

1.2 Significant of study

The increased use of radiation processing for industrial, medical and agricultural applications have motivated research on new materials with adequate dosimetric properties (Li et al., 2005). In this connection, nanostructured materials show unique features that cannot be obtained from conventional macroscopic materials and for this reason interest in nanosized phosphors has grown due to some positive points on detection of high energy ionizing radiations (Kortov, 2010). Their peculiar properties arises from their increased surface to volume ratio and changed the electronic structure due to quantum confinement effect. The surface states are very important to the physical properties, especially the optical properties of the nanoparticles (Sharma et al., 2011). As the particles become smaller the surface to volume ratio and the surface states increase and reduce the excited emission via non-radiative surface recombination. These preliminary remarkable results obtained in such nanomaterials are of great importance to further study the TL properties of different TL nanomaterials for high dose ionizing radiation (Sahare et al., 2007; Salah, 2011). Since, borate compounds possess an effective atomic number which is of human tissue equivalent, they are promising candidates to study and possible use in medical applications and personal dosimeters (Mayles et al., 2007). Calcium borate is one of the suitable and less investigated materials which has enough capability for detecting high-dose ionizing radiation for radiation dosimetry.

1.3 Problem statement

Phosphorous materials have important dosimetric properties such as high sensitivity, a linear response, excellent stability, simple and single glow curve structure ideally around 200 °C, simple annealing procedure for reusability, low fading and non-toxicity. There are several dosimetric materials which are used in industry and many new compounds have been produced by researchers, but none of them has all the above stated properties. Moreover, bulk phosphors materials presented dose-ranges depending on their sensitivity and response characteristics (linearity and saturation) to high-energy radiations. At low doses, very low signal to noise makes it difficult to estimate doses, while at higher doses the saturation of the TL signal dominates (Salah et al., 2011). Therefore, there have always been attempts to either prepare new dosimetric materials with better TL characteristics or simply improve upon the already existing dosimetric materials by varying the concentration of the impurities or by co-doping the phosphor with other elements or doping new impurities in new matrices. Recent studies on nanomaterials opened a new view on improvement and development of luminescent materials with controlled size and shape. Powders with nanometer scale lead to the production of new materials with efficient characteristics and enough capability in dosimetry of ionizing radiations for measurements both low and high doses using the TL technique, where the conventional microcrystalline phosphors saturate (Rivera, 2011; Salah et al., 2009a).

1.4 Scope of study

This research deals with different synthesis conditions, surface modification processes and the thermoluminescence characteristics of a newly developed nanomaterial for radiation dosimetry. In particular, this thesis examines the optical properties of un-doped and doped calcium borate nanocrystals for radiation monitoring in different dosimetry regions from low to high exposures. To produce this new developed nanomaterial a new synthesis approach, co-precipitation technique along with heat treatment, is presented. The co-precipitation technique is capable of producing amorphous, uniform and small sized particles with narrow size distribution and more homogeneity. In continuing; the heat treatment was performed to modify the amorphous particles to nanocrystalline particles for better efficiency in dosimetric applications. The surface modification of particles is carried out by forming an epitaxial organic layer of polyvinyl pyrrolidone surround the particles which showed a good control on the morphology of synthesized nanoparticles after the heating process and an increase in the stability of synthesized particles from agglomeration. The experimental conditions such as concentration of capping agent, annealing temperature and time have great influence on the shape and size of produced nanoparticles. Since the luminescence properties of a materials greatly affected by the characteristics of prepared powders. All the attempts focused on the production of particles with more regular shapes and uniform distribution and enough capability for dosimetric application. The first part of this study investigated a simple method of preparation of TL materials as a new method in development of luminescent materials instead of conventional solid state methods and in continue;

the effect of PVP concentration was monitored by the adjustment of the PVP to water weight ratio from 0 to 3 wt% during the precipitation step and then the produced nanoparticles were subjected to heating process. In the second step, in order to find out the optimal synthesis condition of tetraborate phase structure, different annealing temperatures from 700 to 970 ^{o}C and variety of annealing times from 1 to 5 h were examined. In continue; the fabrication and characterization of doped nanoparticles at optimum annealing temperature and time was performed. In the last part of this study, the dosimetric capability of synthesized un-doped and doped calcium borate nanoparticles in the wide range of gamma exposures was investigated.

1.5 Study objectives

In terms of knowledge creation, the project involves fundamental research into nanostructure synthesis, surface modification, and thermo-luminescent dose response. To accomplish these, the objectives of the present study are as the following:

1. To develop a simple nanosynthesis method for production of luminescence materials different from the conventional solid state methods.
2. To synthesize un-doped and Cu-Mn doped calcium borate nanoparticles for radiation dosimetry application.
3. To investigate the influence of a capping agent on the surface modification, shape and size of synthesized nanoparticles.

4. To monitor the effect of annealing temperatures and annealing times on the phase formation, particle size and size distribution of nanoparticles.
5. To explore the dosimetric capability of synthesized Cu-Mn doped calcium borate nanoparticles as thermoluminecent dosimeter.

1.6 Thesis outline

The structure of this thesis is divided into six chapters. Chapter I, deals with the general introduction about research background, scope, problem statement and objectives of the study. Chapter II, focused on the history of thermoluminescence dosimetry and related literature in view of bulk and nano TLD materials and their TL characteristics on personal, medical and environmental radiation monitoring. The general features and structural properties of borate families, the physical fundamental of thermoluminescence phenomenon and the related theories of TL parameters are presented in chapter III. The methodology of the study, including materials, the experimental details of the synthesis method and description of characteristic technique are described in chapter IV. The subsequent chapter (V) forms the major part of this thesis, in which the experimental results are presented, analyzed, and discussed in details. The last chapter (chapter VI) concludes with a summary and suggestions for future work.

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