UNIVERSITI PUTRA MALAYSIA

STRUCTURAL, THERMAL, OPTICAL AND DIELECTRIC STUDIES ON RARE EARTH ION-DOPED BORATE GLASSES FOR SOLID STATE LASER APPLICATION

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FK 2019 62

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By

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

January 2019

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

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January 2019

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Light emission diodes (LEDs) are widely used in a variety of applications that are available today. With their tunability and long operation life-time, LEDs have surpassed the efficiency and functionality of conventional lighting systems like incandescent and luminescence light sources. Commercial LEDs are typically fabricated using a blue chip coated with yellow phosphor. However, this fabrication method lacks of a red-light component which induces poor thermal stability. A solution to this is to replace phosphors with glass. This work aims to fabricate glass hosts doped with rare earth (RE) elements for better thermal stability and lower power consumption.

Firstly, the singly Dy^{3+}-doped borate glasses with nominal composition (60-x) B_2O_3-10 ZnO-10 PbO-10 Na_2O-10 CaO-(x) Dy_2O_3 (x = 0, 0.1, 0.2, 0.5, 0.75, 1.0, 1.5 and 2.0 mol%) were prepared by using the melt quenching technique which were characterized using X-ray Diffraction (XRD) and Scanning electron microscope (SEM) to confirm the amorphous nature of the glasses and energy dispersive x-ray analysis (EDAX) to validate that all related elements were present in the synthesized glasses. The thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) measurements were also performed to study thermal properties, where $\Delta T > 100$ °C ($\Delta T = T_x - T_g$) for all the glasses. Among all tested Dy^{3+}-doped glasses, 0.75 mol% Dy^{3+}-doped glass showed the highest photoluminescence (PL) intensity with four emissions, where the two transitions corresponded to $^4F_{9/2} \rightarrow {}^6H_{15/2}$ (blue) and $^4F_{9/2} \rightarrow {}^6H_{13/2}$ (yellow) were observed to be more intensified than the others. The Commission International de l'Eclairage (CIE) chromaticity (x,y) coordinates for BZPNCDy 0.1 mol% glass are (0.398, 0.430), close to the white light region in the CIE 1931 chromaticity diagram.

The work was continued with fabricating singly doped Tb^{3+} and Sm^{3+} ions along with co-doped Tb^{3+}/Sm^{3+} borate glasses via melt quenching technique. Both TGA and DSC analysis were conducted to explore the material's thermal properties. Among all Tb^{3+}/Sm^{3+} co-doped glasses, the (Tb0.5-Sm0.5) glass shows the highest emission intensity with respect to others. A total of five emission bands were found, where two were from Tb^{3+} transitions corresponding to 488 nm (blue) ($^5D_4 \rightarrow {}^7F_6$) and 543 nm (green) ($^5D_4 \rightarrow {}^7F_5$). Three emission bands for Sm^{3+} at 563 nm (green), 599 nm (orange-red) and 645 nm (red) according to $^4G_{5/2} \rightarrow {}^6H_{5/2}$, $^4G_{5/2} \rightarrow {}^6H_{7/2}$, and $^4G_{5/2} \rightarrow {}^6H_{9/2}$ electronic transitions have been identified. The calculated CIE chromaticity (x,y) coordinates for singly doped Tb^{3+} (Tb0.5) green emission, singly doped Sm^{3+} (Sm0.5) orange-red emission, and co-doped Tb^{3+}/Sm^{3+} (Tb0.5-Sm0.5) yellow emission are (0.343, 0.584), (0.607, 0.389), and (0.438, 0.515), respectively, following the CIE 1931 chromaticity diagram.

Abstrak tesis ini dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

KAJIAN SIFAT STRUKTUR, TERMA, OPTIKAL DAN DIELEKTRIK PADA ION TERDOP NADIR BUMI KACA BORATE UNTUK APLIKASI LASER KEADAAN PEPEJAL

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Diodes pelepasan cahaya (LED) digunakan secara meluas dalam pelbagai aplikasi yang tersedia hari ini. Dengan kebolehan untuk menala serta mempunyai jangka masa operasi yang panjang, LED telah melepasi kecekapan dan fungsi sistem lampu konvensional seperti sumber lampu pijar dan pendarfluor. LED komersial biasanya direka menggunakan cip biru yang disalut dengan fosfor kuning. Walaubagaimanapun, kaedah fabrikasi ini tidak mempunyai komponen cahaya merah yang mendorong kestabilan terma yang lemah. Satu penyelesaian untuk ini adalah untuk menggantikan fosfor dengan kaca. Kerja penyelidikan ini bertujuan untuk mengarang tuan rumah kaca yang disebat dengan unsur-unsur nadir bumi (RE) untuk kestabilan haba yang lebih baik dan penggunaan kuasa yang lebih rendah

Ringkasan penyelidikan terbahagi kepada dua bahagian. Bahagian yang pertama dari gentian kaca borate Dy^{3+} dengan komposisi nominal (60-x) B_2O_3-10 ZnO-10 PbO-10 Na_2O-10 CaO- (x) Dy_2O_3 (x = 0, 0.1, 0.2, 0.5, 0.75, 1.0, 1.5 dan 2.0 mol%) telah disediakan menggunakan teknik pencairan pelindap kejutan. Biasan Sinar-X (XRD) dan Pengimbasan Mikroskop Elektron (SEM) mengesahkan sifat samar-samar kaca melalui Spektroskopi Penyebaran Tenaga (EDAX), dimana semua elemen yang berkaitan berjaya ditemui dalam kaca yang disintesis. Pengukuran Termogravi Metrik (TGA) dan Pegimbasan Perbezaan Kalorimetri (DSC) dilakukan untuk mengkaji sifat terma, di mana $\Delta T > 100$ °C ($\Delta T = T_x - T_g$) untuk semua gentian kaca. Di antara semua gentian kaca Dy^{3+}, gentian kaca 0.75 mol% Dy^{3+}-menunjukkan intensiti Fotoluminesensi (PL) tertinggi dengan empat tahap pelepasan, di mana dua peralihan sepadan dengan $^4F_{9/2} \rightarrow {}^6H_{15/2}$ (biru) dan $^4F_{9/2} \rightarrow {}^6H_{13/2}$ (kuning) diperhatikan lebih tinggi daripada yang lain. Suruhanjaya Internationale de l'Eclairage (CIE) mengklasifikasikan koordinat kromatik (x, y) untuk BZPNCDy 0.1 mol% adalah

(0.398, 0.430), hampir dengan medan cahaya putih dalam gambarajah kromatik CIE 1931.

Di bahagian kedua, ion Tb^{3+} tunggal dan ion Sm^{3+} yang disambungkan bersama-sama dengan gentian kaca Tb^{3+}/ Sm^{3+} borate direka oleh teknik pencairan pelindap kejutan. Kedua-dua analisis Termogravimetrik (TGA) dan Pengimbasan Kalorimetri Berbeza (DSC) diukur untuk mengkaji sifat terma. Di antara semua gentian kaca Tb^{3+}/ Sm^{3+}, kaca (Tb0.5-Sm0.5) menunjukkan keamatan emisi tertinggi dibandingkan dengan yang lain. Sejumlah lima julat pelepasan di mana dua dari perpindahan Tb^{3+} sepadan dengan 488 nm (biru) ($^5D_4 \rightarrow {}^7F_6$) dan 543 nm (hijau) ($^5D_4 \rightarrow {}^7F_5$) dijumpai. Tiga julat pelepasan untuk Sm^{3+} pada 563 nm (hijau), 599 nm (oren merah) dan 645 nm (merah) mengikut $^4G_{5/2} \rightarrow {}^6H_{5/2}$, $^4G_{5/2} \rightarrow {}^6H_{7/2}$ dan $^4G_{5/2} \rightarrow {}^6H_{9/2}$ peralihan elektronik dikenalpasti. Keputusan CIE (x, y) koordinat untuk Tb^{3+} (Tb0.5) tunggal pelepasan hijau, sm^{3+} (Sm0.5) tunggal pelepasan oren merah dan dwi-gentian Tb^{3+}/ Sm^{3+} (Tb0.5-Sm0.5) pelepasan kuning adalah masing-masing (0.343, 0.584), (0.607, 0.389), dan (0.438, 0.515), mengikut gambarajah kromatik CIE 1931.

ACKNOWLEGEMENTS

I would like to thank my father Fadhil Abbas for his supported during all studding times, calls, and blessings. I am also thankful to my mother for her dedication, brother, friend and many others for their support.

In the beginning, I would like to express my sincere gratitude to my supervisor, Dr. Gandham Lakshminarayana, for his support throughout my Ph.D. study. You have definitely provided me with the tools that I needed to choose the right direction and successfully complete my thesis.

I am grateful to Dr. Mohd Adzir bin Mahdi, for enlightening me the first glance of research and monitoring my Ph.D. direction and related research. I would like to thank him for his patience, motivation, and immense knowledge.

I would like to express my sincere gratitude to my supervisory committee members, Dr. Sharudin Bin Omar Baki, for his constructive suggestions during my research period and for supporting me through my studying time.

Finally, I am thankful with love and respect to my wife, Zeena Najeeb. She has been my best friend and great companion, loved, supported, encouraged, entertained, and helped me get through this agonizing period in the most positive way. I would like to dedicate this thesis with love to my son Ali, and the new baby girl Mila.

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

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This is to confirm that:

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LIST OF ABBREVIATIONS

Δ	Glass Stability Factor
ΔE	Energy Gap
ATR-FTIR	Attenuated Total Reflectance-Fourier Transform Infrared
CR	Cross Relaxation
D.W.	Distilled Water
DSC	Differential Scanning Calorimetry
DWDM	Dense Wavelength Division Multiplexing
Dy^{3+}	Dysprosium ions
ED	Electric Dipoles
EDAX	Energy dispersive X-ray analysis
Er^{3+}	Erbium ions
ESA	Excited State Absorption
ET	Energy Transfer
ETU	Energy Transfer Up conversion
ε'	Dielectric constant
ε''	Dielectric loss
FWHM	Full-Width at Half-Maximum
GSA	Ground State Absorption
H_R	Huruby's Parameter
I_{em}	Light Intensity
J	Total Angular Momentum
JO	Judd-Ofelt
LD	Laser Diode

M	Metallization Criterion
MD	Magnetic Dipoles
MW	Molecular Weight
NBO	Non-Bridging Oxygen
NIR	Near Infrared
Ø	Bridging Oxygen
OPD	Oxygen Packing Density
p	Phonon Energy
PL	Photo Luminescence
PLE	Photo Luminescence Excitation
RE^{3+}	Rare Earth ions
RL	Reflection Loss
Rm	Molar Refractivity
$S_{calc.}$	Theoretical Electric Dipole Line Strength
$S_{ed.}$	Electric Dipole Line Strength
SEM	Scanning electron microscopy
Sm^{3+}	Samarium ions
$S_{meas.}$	Measured Line Strength
SSL	Sold-State Laser
Tb^{3+}	Terbium ions
TDA	Differential Thermal Analysis
T_g	Transition Temperature
TGA	Thermo-Gravimetric Analysis
T_p	Melting Temperature
TPA	Two-photon absorption

TTT	Time-Temperature-Transformation
T_x	Crystallization Temperature
UCL	Upconverted Luminescence
UV	Ultra Violet
VIS	Visible
V_M	Molar Volume
WDM	Wavelength Division Multiplexing
W_{nr}	Nonradiative Rate
Xi	Pauling Electronegativity
XRD	X-ray Diffraction
$\alpha(\lambda)$	Optical Absorption Coefficient
αm	Molar Polarizability
Γ	Orbital Angular Momentum
η	Quantum Efficiency
$\rho(v)$	Radiation Density
ρ_L	Liquid Density
σ	AC conductivity
$\tau_{exp.}$	Measured Lifetimes
τ_{rad}	Radiative Decay Lifetimes
Ω_λ	JO Intensity Parameters

CHAPTER 1

INTRODUCTION

1.1 General introduction

Glass is a well-known amorphous material used in many structural applications for the past few centuries. However, it was not until the twentieth century that the impact of glass took a new perspective, as advanced electronics like the radio, television and smartphones came about. This also pushed scientists to explore new compositions of glass to attain superior electrical properties and fit the current commercial demand. Example of widely used glass compositions today are oxide-based glasses like silicates, borates, phosphates, or germanates, (SiO_2, B_2O_3, P_2O_5, or GeO_2) [1-10]. Another notable glass composition that has gained the interest of many physicists and engineers are rare-earth (RE) doped glasses. The 4f electronic configuration of RE ions in varied glass matrixes made emissions from ultraviolet to infrared possible which had proliferated into many potential applications including display devices, optical fiber amplifiers, high-intensity optical devices, optical information processing, optoelectronic devices, non-invasive temperature sensors, and solar cells [11-17]. In laser technology, the RE-doped glasses have been considered as good luminescence materials for their application in solid-state lasers (SSL) following their visible emissions, facile manufacturing processes, and good thermal stability [17-23]. In the present study, a wide range of oxide-based borate glass compositions were synthesized and selected for rare-earth doping. The selection of materials is based on the physical characteristics as glass modifier/intermediates mentioned in the literature review. However, higher phonon energy (~1300–1500 cm-1) demerits that lead to restrict their applications because of the non-radiative transitions. With an inclusion of appropriate network modifier oxide Na2O and heavy metal PbO oxide, phonon energy of the borate glass can be reduced for the higher quantum efficiency. The addition of ZnO into the borate network gives an advantage as non-hygroscopic nature, and non-toxicity, which in turn increases the possibility for optoelectronic applications. As well, it is established that ZnO have the ability of penetrate the glass network structure as a modifier or network former depending on the ZnO molar concentration. Calcium borate glasses are very attractive to evaluate the effects of the chemical environment on the optical properties of RE ions by contributing to low melting temperature, high thermal stability and non-hygroscopicity [18]. Solid stat lighting available commercially like phosphor-converted white LEDs (pc-WLEDs) are manufactured by the Y3Al5O12: Ce3+ (YAG:Ce) yellow phosphor integration together with blue LEDs as result of low cost LEDs. The YAG:Ce- type white LEDs emitting cool white light, and their application is limited according to low color rendering index (CRI < 80) and high correlated color temperature (CCT = 4,000–8,000 K) [20-24].

1.2 Problem statement

Solid state lights that are commercially available today, like phosphor-converted white light emitting diodes (pc-WLEDs), are manufactured by integrating yellow phosphor; Y3Al5O12: Ce3+ (YAG:Ce); with a mixture of green, blue and red phosphors excited using a ultra-violet LED chip [24-27]. However, this method induces low thermal quenching temperature which causes high correlated color temperature (4,000 – 8,000 K) and low color rendering index (CRI < 80) that may result to inefficiency, high power consumption and unnecessary cost [28-36]. Hence, there is a need to find more suitable materials that can provide highly efficient and cost-effective solid-state lighting to address the problems. Through RE ions doped materials, the Tb^{3+} and Sm^{3+} co-doped glasses are considered for their special spectral characteristics, emission intensity and energy transfer from Tb^{3+} to Sm^{3+} [37-41].

1.3 Objectives of the study

In this thesis, mainly borate glass in the composition B_2O_3-ZnO-PbO-Na_2O-CaO doped with Dy^{3+} (singly) and co-doped Sm^{3+}/Tb^{3+} ions luminescence in the visible spectral region is reported. The main objectives of this thesis can be concluded as:

(i) To investigate borate glass as host glass doped with RE-ions for solid-state lighting (SSL) applications.
(ii) To fabricate and characterize the singly doped and co-doped RE-ions (different concentrations) in borate glass as host matrix, through the structural, thermal, optical, and dielectric features.
(iii) To propose both RE-ions doped and co-doped borate glasses as potential candidates for SSL applications, by exploring them in terms of spectroscopic properties.

1.4 Outlines of the Thesis

The six chapters of the present thesis are outlined as follows:

Chapter 1 presents general introduction optical materials based on oxide glasses and their different applications. Additionally, the problem statement and the main objectives of the work are also mentioned.

Chapter 2 reviews the principle of glass forming. Glass formers, which play an important role for optical materials (e.g., glasses), reviewed in this chapter, are borate and silicate glasses. In this part, the reason for choosing borate as a glass former in order to obtain the research objectives is explained.

Chapter 3 introduces RE^{3+} elements and their function in borate glass hosts. In the beginning, the basic RE spectroscopic theory is presented, which accounts for the observed absorption spectra of the RE ions in solids. This is followed by the luminescence of RE ions and demonstration of hypersensitive transitions along with transition probabilities, Cross relaxation (CR) of ions mechanisms and Concentration quenching effect in RE ions. Moreover, energy transfer and ions interaction are explained for RE^{3+} doped host glasses.

Chapter 4 highlights the glass sample preparation and method of characterization. The detailed steps of important preparation procedure are described here, which begins with glass sample calculations, followed by batching procedure, and glass fabrication.

Chapter 5, investigates the suitable host for SSL applications by using single doped and co-doped rear earth ions Dy^{3+}, Tb^{3+}/ Sm^{3+}, respectively, which was divided into two sections. Section 5.1 presents the structural, thermal, optical and dielectric properties of borate glass introduced with Dy^{3+}-doped at different concentrations, as a result, it shows that all those glasses have similar structural, thermal optical and dielectric properties. On the other hand, Section 5.2 presents Tb^{3+}/ Sm^{3+} co-doped borate glasses at deferent concentrations. In this section, thermal optical energy transfer and dielectric properties are presented.

Chapter 6 concludes the findings of the thesis. The results of all systematic objectives are presented in order to obtain a suitable host glass for solid stat lighting applications.

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LIST OF PUBLICATIONS

In this part of thesis, presenting the research papers below, which published based on the thesis results and related works.

Thesis results

Al-BFA Mohammed, Lakshminarayana, G., Baki, S. O., Halimah, M. K., Kityk, I. V., & Mahdi, M. A. "Structural, thermal, optical and dielectric studies of Dy^{3+}: B_2O_3-ZnO-PbO-Na_2O-CaO glasses for white LEDs application," *Optical Materials*, vol. 73, pp. 686-694, 2017.

Al-BFA Mohammed, Lakshminarayana, G., Baki, S. O., Kh. A. Bashar, Kityk, I. V., & Mahdi, M. A. "Optical and dielectric studies for Tb^{3+}/Sm^{3+} co-doped borate glasses for solid-state lighting applications," *Optical Materials*, vol. 86, pp. 387-393, 2018.

Related works

Bashar, Kh A., G. Lakshminarayana, S. O. Baki, Al-BFA Mohammed, U. Caldiño, A. N. Meza-Rocha, Vijay Singh, I. V. Kityk, and M. A. Mahdi. "Tunable white-light emission from Pr^{3+}/Dy^{3+} co-doped B_2O_3-TeO_2 PbO-ZnO Li_2O-Na_2O glasses," *Optical Materials*, vol. 88, pp. 558-569, 2019.

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