STRUCTURAL AND OPTICAL PROPERTIES OF PEKOVITE AND MALEEVITE PHOSPHOR FOR WHITE LED APPLICATIONS

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DEDICATION

This thesis is dedicated to

my beloved father (Leow Bin Huat) and mother (Lai Yook Kuen)

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ABSTRACT

Polycrystalline pekovite ($SrB_2Si_2O_8$) and maleevite ($BaB_2Si_2O_8$) phosphor doped with Eu and/or Dy ions of nominal composition (25-*a*-*b*)MO-25B_2O_3-50SiO_2: *a*Eu_2O_3, *b*Dy_2O_3 where M = (Sr, Ba) and *a*, *b* = 1 or 0 mol% were synthesized via solid state reaction to study their structural and optical properties for white LED applications. The maleevite and pekovite phosphor were sintered at temperature of 950 and 1000 ^{o}C respectively for 5 hours in air and CO. The XRD results revealed that the incorporations of dopants caused the mean crystallite sizes to increase from 18.01 – 115.93 nm indicating improved crystallinity which is in orthorhombic structure and this was confirmed by SEM. The rare earth dopants were incorporated in the crystal and were confirmed by EDAX. FT-IR and FT-RAMAN spectra revealed the presence of borosilicate superstructure. Both danburite breathing mode along with multiple vibrational modes in FT-IR were found to be red-shifted when strontium were replaced with barium due to the larger ionic radii which increased Ba-O bond length. UV-VIS-NIR diffused reflectance spectra were collected and the optical band gaps of the samples were determined which were in the ranges 3.23 – 4.25 eV in pekovite and 3.42 – 3.7 eV in maleevite samples. Photoluminescence spectra of Eu^{3+} and Dy^{3+} ions were obtained from UV excitation which showed intense emission peaks at 483 and 575 nm from Dy^{3+} along with 590 and 613 nm from Eu^{3+} and the dopant site symmetry ratios were calculated. Combinations of blue, green and red emission from Eu^{3+} and Dy^{3+} showed its potential as a tune-able white light phosphor. Emissions at ~435 nm from Eu^{2+} were detected in all CO sintered phosphor and air sintered pekovite, exhibiting the unusual reduction of Eu^{3+} to Eu^{2+} in air. Overall, low cost phosphor materials which would be suitable for white LED applications were synthesized.

ABSTRAK

Fosfor polihablur pekovite ($SrB_2Si_2O_8$) dan maleevite ($BaB_2Si_2O_8$) didop dengan ion-ion Eu dan/atau Dy dengan komposisi nominal (25-*a*-*b*)MO-25B_2O_3-50SiO_2: *a*Eu_2O_3, *b*Dy_2O_3 dimana M = (Sr, Ba) dan *a*, *b* = 1 atau 0 mol% telah disintesis dengan kaedah tindak balas keadaan pepejal untuk mengkaji sifat struktur dan optik fosfor bagi aplikasi LED cahaya putih. Fosfor maleevite dan pekovite telah disinter masing masing pada suhu 950 dan 1000 ^{o}C selama 5 jam dalam udara dan CO. Keputusan XRD menunjukkan apabila ion dopan meresap dalam struktur hablur, saiz hablur purata meningkat daripada 18.01 kepada 115.93 nm dan menunjukkan peningkatan penghabluran yang berbentuk orthorhombic sebagaimana ditunjukkan oleh SEM. Resapan ion dopan ke dalam hablur dibuktikan oleh EDAX. Spektra FT-IR dan FT-RAMAN menunjukkan kehadiran struktur-super borosilikat. Kedua-dua mod getaran mampatan dan regangan danburite dan pelbagai mod getaran dalam FT-IR didapati berubah ke panjang gelombang yang lebih panjang apabila strontium diganti dengan barium yang mempunyai jejari ion yang lebih besar yang menambahkan panjang ikatan Ba-O. Spektra kepantulan terbaur UV-VIS-NIR dirakam dan jurang jalur optik sampel ditentukan dimana nilainya dalam julat 3.23-4.25 eV untuk pekovite dan 3.42-3.7 eV untuk maleevite. Spektra kefotopendarcahayaan ion Eu^{3+} dan Dy^{3+} yang diuja dengan cahaya UV mempamerkan puncak pancaran terang pada 483 nm, 575 nm dari Dy^{3+} dan 590 nm, 613 nm dari Eu^{3+} kemudian nisbah simetri tapak dopan dihitungkan. Gabungan pancaran biru, hijau dan merah daripada Eu^{3+} dan Dy^{3+} mempunyai potensi sebagai fosfor cahaya putih boleh-laras. Puncak pancaran Eu^{2+} pada ~435 nm dikesan dalam semua fosfor yang disinter dalam CO dan juga pekovite yang disinter dalam udara, menunjukkan penurunan luar biasa Eu^{3+} kepada Eu^{2+} dalam udara. Secara keseluruhanya, bahan fosfor yang murah dan sesuai digunakan dalam aplikasi LED cahaya putih telah disintesis.

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LIST OF ABBREVIATIONS

BAM	-	$BaMgAl_{10}O_{17}$
BSE	-	Back scattered electrons
CIE	-	Commission International del'Eclairage
CO	-	Carbon Monoxide
COD	-	Crystallography Open Database
CRT	-	Cathode ray tube
CT	-	Charge transfer
CTB	-	Charge transfer band
DRS	-	Diffused reflectance spectra
EDX	-	Energy dispersive X-ray
et al.	-	et alia (and others)
etc.	-	et cetera (and other things)
FL	-	Fluorescent lamp
FT	-	Fourier transform
FTIR	-	Fourier transformed infrared
FHWM	-	Full width half maximum
GOF	-	Goodness-of-fit
HDTV	-	High definition television
HOMO	-	Highest occupied molecular orbital

ICDD	-	International Centre for Diffraction Data
IR	-	Infrared
KM	-	Kubelka-Munk
LCD	-	Liquid crystal display
LED	-	Light emitting diode
LUMO	-	Lowest unoccupied molecular orbital
MQW	-	Multiple quantum well
NBO	-	Non-bridging oxygen
NIR	-	Near-infrared
PDP	-	Plasma display panel
PL	-	Photoluminescence
RGB	-	Red/Green/Blue
SEM	-	Scanning electron microscope
SSL	-	Solid state lighting
TV	-	Television
UV	-	Ultraviolet
Vis	-	Visible
VUV	-	Deep ultraviolet
WLED	-	White light emitting diode
YAG	-	$(Y,Ga)_2Al_5O_{12}$

LIST OF SYMBOLS

$\%T$	-	Transmittance
μ	-	Reduced mass
ε	-	Absorptivity
η_T	-	energy transfer efficiency
θ	-	Diffracted angle
λ	-	Wavelength
χ^2	-	Goodness of fit
A	-	Absorbance
a	-	Eu_2O_3 dopant concentration (mol%)
b	-	Dy_2O_3 dopant concentration (mol%)
c	-	Velocity of light
d	-	Spacing between planes
D	-	Diffusion coefficient
D_o	-	Maximum diffusion coefficient at infinite temperature
E	-	Energy
E_A	-	Activation energy for diffusion
h	-	Plank’s constant
I	-	Samples spectrum intensity
$I(\lambda)$	-	Spectral power distribution

I_o - Background spectrum intensity

Is - Peak intensity of the sensitizer in the presence of acceptor

I_{so} - Peak intensity of the sensitizer in the absence of acceptor

k - Force constant of a bond

l - Thickness

m - Mass

M - Sr or Ba

n - Integer value

R - Gas constant

R_∞ - Reflectance of an infinitely thick sample

S - Intensity ratio

T - Temperature

v - Frequency

x, y - chromaticity coordinate

$\bar{x}, \bar{y}, \bar{z}$ - standard observer color matching functions

X, Y, Z - Tristimulus values

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CHAPTER 1

INTRODUCTION

2.1 Background of Study

The development of white light emitting diode (LED) had drawn increasing interest in the past few years for its potential applications in solid state lighting i.e. flat panel display and illuminations systems (Ma et al. 2012, Tian et al. 2010, Horng et al. 2007, Xie et al. 2010). Research works on solid state lighting used to be focused around fluorescent tubes. These tubes functions based on the conversion of 254 nm mercury vapor lamp into visible light using phosphor. However, mercury vapor lamp based lighting is slowly being replaced by a more efficient lighting system of LEDs. A considerable amount of studies revolving LED were carried out majorly due to its high energy efficiency, long usable life time, compact size and environmental friendliness (Li et al. 2011, Wang et al. 2014a).

Nowadays, commercially available white LEDs were produced from InGaN-based LED chips coated with red/green/blue (RGB) phosphor (Yang et al. 2007, Sharma and Towe 2010). However, such white LED suffers a major drawback in low color rendering index due to the lack of red component (Que et al. 2013). Widely available $(Y,Gd)BO_3:Eu^{3+}$ red phosphor has a chief disadvantage in low efficiency (Xiaoxia et al. 2009). Not to mention the distinct emission of Dy^{3+} in the blue and green regions can serve as a key component for white light production. Much effort could be reduced by a readily white emitting phosphor without needing to combine the RGB such as from a Dy^{3+} doped phosphor. Despite the high preparation temperature (>1400 ^{o}C), current commercially available blue phosphor

$BaMgAl_{10}O_{17}:Eu^{2+}$ (BAM) also has deficiency in UV absorption (Won et al. 2010, Deng et al. 2013).

With the introduction of AlGaN and InGaN LED chips, the band gap of the devices can be tuned from 3.2 to 6 eV by varying the content ratio of Al/In to GaN (Horng et al. 2007, Sharma and Towe 2010, Yang et al. 2007). This in turn produces emissions from 200 to 400 nm wavelength that benefits rare earth based phosphor for its ability to be efficiently excited in the UV regions. The wide excitations of rare earth based phosphor also had a small difference in energy between excitation and emission wavelength that promotes energy efficiencies. Another key interest in rare earth dope systems were the energy transfer between dopant ions within the host system. Many a times in a co-doped phosphor, a dopant could act as a sensitizer that absorbs excitation energy and transfer it over to the acceptor. These mechanisms usually resulted in an enhanced emission of the acceptor therefore increases a phosphor's luminescence performance and energy efficiency.

One key important criterion for a white LED to be produced for commercial use is its ability to adjust its white light temperature by means of varying the chemical composition of the host material, dopants or dopants concentrations. Commonly available white LED from the combination of blue light from LED chip and yellow light of $(Y, Ga)_3Al_5O_{12}:Ce^{3+}$ (YAG:Ce) phosphor produced a cool white light. The white light produced were more bluish in relative to conventional incandescent and fluorescent lamps (Aboulaich et al. 2014). These devices lack the red component and therefore have a poor color rendering index. For general light purposes, the white light needs to be shifted toward the orange/red wavelength region to produce a 'warmer' light.

Borosilicate glass or ceramic system had been the center of interest in many applications including laboratory glassware and optical glass, mainly due to its chemical durability and abundance of SiO_2 while attaining a low temperature synthesis due to borate as modifier (Sembiring 2011, Zawrah and Hamzawy 2002). Moreover, further investigation into alkaline earth borosilicate system had been a field of interest for its potential in application such as low cost optical connectors, optical amplifiers and phosphor materials (Rao et al. 2014b, Jean and Chang 1999). Also, applications of low-temperature co-fired ceramics (LTCC) of alkaline earth borosilicate in wireless communication and microwave products has recently immerged (Chen et al. 2013, Krzhizhanovskaya et al. 2010). One of the naturally occurring alkaline earth borosilicate minerals were the danburites ($CaB_2Si_2O_8$). Maleevite ($BaB_2Si_2O_8$) and pekovite ($SrB_2Si_2O_8$) were analogous species to danburite where the Ca^{2+} cations are replaced by Sr^{2+} and Ba^{2+} respectively. $SrB_2Si_2O_8$ and $BaB_2Si_2O_8$ is a good candidate for phosphor host materials owing to its colorless transmitted light, clarity, hardness (Mohs hardness = 7) as well as chemically and physically stable properties (Pautov et al. 2004). As far as cost is concerned, the abundance of precursor material of B_2O_3, $BaCO_3$ and SiO_2 together with the low sintering temperature of below 1000 ^oC give researchers and manufacturers an edge on cost effectiveness. Moreover, reports of unusual reduction of trivalent Eu ions to divalent oxidation state in various systems had been reported by several researchers before. The reduction provides the desired formation of Eu^{2+} ions in the host system without employing sophisticated equipment to control the sintering atmosphere.

On the other hand, conventional fabrication method by solid state reaction is adapted by many works on luminescent materials. It is found to be easy method and allows crystal forming well below melting point. This technique involves dry pressing with high pressure on the mixed sample powder which produces the so called “green body”, which is usually sintered at temperature 2/3 of melting point for several hours. For instance $Ca_3MgSi_2O_8$:Eu,Dy were synthesized via sintering at 1200^oC for 4 hours and MgO-SiO_2-P_2O_5:Eu,Dy phosphor were prepared via sintering at 900^oC for 4 hours (Musdalilah 2011, Lin et al. 2001). Consequently, the polycrystalline solids are formed by this method which is also known as ceramics. Ceramics are generally non-metallic and inorganic solid that are crystallized or partly crystallized. The weak luminescence properties of amorphous solid material set limits to their application in optical devices due to glass nature of being translucent. (Cheng et al. 2009).

2.2 Statement of Problem

Syntheses of pekovite were firstly performed by Verstegen et. al in 1972 (Verstegen et al. 1972). Quite recently, several researchers had reported on $Sr_3B_2SiO_8$, $SrB_2Si_2O_8$, and $BaB_2Si_2O_8$ crystals for its potential in phosphor applications and showed promising result. (Wang and Wang 2011, Wang et al. 2009, Saradhi et al. 2010, Sabikoglu 2013). Saradhi et. al. and Wang et. al. reported rare earth activated $SrB_2Si_2O_8$ and $BaB_2Si_2O_8$ that exhibited strong UV absorption. (Wang et al. 2009, Saradhi et al. 2010). Rare earth doped phosphor has always been of significant interest for its long wavelength excitation in UV. This feature is important for application in fluorescent lighting, plasma display phosphor, LED, and bills marking. Considering the fact that a considerable amount of research had been performed on these phosphors, the structural and luminescence investigations on $SrB_2Si_2O_8$ $BaB_2Si_2O_8$ crystals were very limited. To the best of our knowledge, Eu and Dy co-activated in $SrB_2Si_2O_8$ and $BaB_2Si_2O_8$ host crystal had not been assessed before. Also, based on extensive review in literature, the structural and optical properties between $SrB_2Si_2O_8$ and $BaB_2Si_2O_8$ had not been compared in studies before.

As mentioned earlier, the red phosphor of (Y,Gd)BO3:Eu^{3+} were low in energy efficiency. The lack of efficient red phosphor hampered the color rendering index of white LED that were based on only blue and yellow emissions. To overcome this, the search for a more efficient red phosphor is needed. Not to mention, the low absorption in the UV region of BAM blue phosphor. Widely available BAM phosphor also had a preparation temperature that is costly. As stated later in this dissertation, presently available phosphors were deficient in many aspects and needed to be addressed. With these motives in mind, the urge to discover a novel, inexpensive yet efficient phosphor is needed.

This current work investigated the structural and luminescence properties of rare earth (Eu^{2+}, Eu^{3+} and Dy^{3+}) doped $SrB_2Si_2O_8$ and $BaB_2Si_2O_8$ for phosphor applications. The specimens will be prepared via solid state reaction method and under oxidizing atmosphere and reducing atmosphere to study the effects of sintering atmosphere. The effects of co-doping/doping Eu and Dy on the structural and luminescence properties were also focused in this study. This study will also compare the result obtained from $SrB_2Si_2O_8$ and its analogous species $BaB_2Si_2O_8$ to study the effects of different alkali earth metal. Reports on the emission color will be presented thoroughly in order to investigate the feasibility of these phosphors for solid state lighting applications.

2.3 Objectives of Study

The objectives of this study are:

1. To synthesize Eu and Dy doped/co-doped $SrB_2Si_2O_8$ and $BaB_2Si_2O_8$ phosphor materials.
2. To determine the influence of sintering atmosphere on the structural, morphology and optical properties of $SrB_2Si_2O_8$ and $BaB_2Si_2O_8$ phosphor.
3. To determine the correlation between doping or co-doping of Eu and Dy on the structural, morphology and optical properties of $SrB_2Si_2O_8$ and $BaB_2Si_2O_8$ phosphor.
4. To investigate the difference between $SrB_2Si_2O_8$ and $BaB_2Si_2O_8$ host systems doped with Eu and Dy in terms structural and optical properties.

2.4 Scope of Study

Based on the many advantages that the maleevite and pekovite minerals deliver, the phosphor material host system in this study was intended to be $SrB_2Si_2O_8$ and $BaB_2Si_2O_8$ compounds. The compounds were to be synthesized by means of dry pressing and solid state reaction method from starting materials SiO_2, H_3BO_3, $SrCO_3$ and $BaCO_3$ in powdered form. Two atmospheres will be allowed in the sintering vicinity: oxidizing atmosphere of air and reducing atmosphere of carbon monoxide (CO). The phosphor will be doped with Eu and Dy to study its influence on the optical and structural properties of the host system. Eu_2O_3 and Dy_2O_3 in powdered form will be used to incorporate the desired dopants.

The crystallinity and phase of the phosphor will be verified by X-ray diffraction. To further study the structure of the phosphor, Fourier Transform Infrared spectroscopy (FTIR) and RAMAN shift spectroscopy will be used to interpret the molecular group in the host system. Scanning electron microscope (SEM) will be employed to investigate the morphology of the phosphor. While the energy dispersive X-ray (EDX) will be used to verify the incorporation of dopants and existence of any undesired impurities. The phosphor samples will be investigated by UV-Vis-NIR spectroscopy for its optical absorption, which in turn will provide information regarding its optical band gap. Photoluminescence spectroscopy (PL) will be used to study the luminescence emission and excitation of the Eu and Dy doped phosphor. Subsequently the emission spectra will be used to generate the Commission International del'Eclairage (CIE) color coordinates to obtain their practical emitting colors.

2.5 **Significance of Study**

This study aims to develop phosphor based on alkaline earth borosilicate for its structural feature and potential luminescent properties. This study will also produce new phosphor material with violet, blue, red and white emission that will solve the energy efficiency lacking red phosphor issue, an alternative blue phosphor for costly BAM phosphor and also readily white emitting phosphor with different 'warmness'. This study also strives to provide phosphor with good luminescence feature such as bright fluorescent and efficient excitation in the UV range which will provide vast application in solid state lighting especially white LEDs. The broad excitation wavelength of these phosphors will provide LED researchers with phosphors that can be excited effectively by the widely available GaN-based LED chips.

This study will also create new phosphors which are easy and low cost to produce, yet lead-free. Anyhow, this research will deliver valuable contribution of knowledge and information on the structural and optical properties of $SrB_2Si_2O_8$ and $BaB_2Si_2O_8$ compounds.

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