STRUCTURAL, ELASTIC AND OPTICAL PROPERTIES OF ZINC-ALUMINO-BOROSILICATE DOPED GADOLINIUM OXIDE GLASS-CERAMICS

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By

NUR ATIKAH NAZIHAH BINTI ISMAIL

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

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DEDICATIONS

To my loving parents,
For their unconditional love and support
To accomplish my research with confidence

To my siblings and family
For making my life complete

To my husband and his family
For constant source of support

To all my very wonderful friends
For making my life full of joy and happiness

To all my lecturers
For helping and guiding me to complete this study

Thank you all

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

STRUCTURAL, ELASTIC AND OPTICAL PROPERTIES OF ZINC-ALUMINO-BOROSILICATE DOPED GADOLINIUM OXIDE GLASS-CERAMICS

By

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In recent years, researchers are developing a great interest towards the fabrication and synthesizing willemite (Zn_2SiO_4) glass-ceramics. However, intensity luminescence of willemite based glass ceramic doped with gadolinium oxide (Gd_2O_3) is less reported. Hence, this study is focusing on fabricate and synthesized willemite based glass-ceramics using Gd_2O_3 as dopant. The Gd_2O_3 doped zinc-alumino-borosilicate (ZnO-Al_2O_3-B_2O_3-SiO_2) glass system were synthesized via the melt-quenching approach using composition $(60–x)ZnO–5Al_2O_3–15B_2O_3–20SiO_2{:}x(Gd_2O_3)$($x$ = 0, 0.5, 1.0, 2.0 and 3.0 mol%) and willemite-based glass-ceramics were obtained from these precursor glasses through a controlled crystallization process.

According to all analysis, the best willemite based glass-ceramic has been selected from the glass system when doped with 3 mol% of Gd_2O_3 and temperature at 800 °C. XRD and FESEM methods were also used to analyze the structural properties of precursor glass, the formation of willemite crystal phase, shape, and size as crystallization temperatures increased. The average approximated crystallite size determined by XRD is in the 50-70 nm range. The structural properties of glass and glass-ceramics were assessed using FTIR spectroscopy. The infrared spectra studies reveal the existence of SiO_2 and ZnO_4 vibrational groups indicating the establishment of the willemite crystal phase. As for elastic analysis, the values for experimental elastic moduli were obtained from ultrasonic velocities measurement by using the non-destructive ultrasonic technique. The longitudinal and shear velocities vary from 4798 to 6976 m/s and 2991 to 3082 m/s, respectively. The experimental elastic moduli (longitudinal modulus (L), shear modulus (G), bulk modulus (K) and Young's modulus (E)) increases from 94.60 to 202.98 GPa, 36.77 to 39.61 GPa, 45.56 to 150.17 GPa and 86.94 to 109.22 GPa, respectively. In addition, the optical band willemite based glass-ceramics doped with Gd_2O_3 fluctuate from 3.64 to 3.38 eV due to structural rearrangement of network. The emission spectra of gadolinium ions show the strong emission peak at wavelengths of 425, 447, 462, 485, and 530

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nm. The willemite phase shows prominent green emission spectra located at 530 nm. These emission spectra produce when the dopant content and heat treatment temperatures increase, the luminescence performance of the glass-ceramics also improves. The incorporation of gadolinium ions into the willemite crystals as heat treatment temperatures increase affect the intensity of the emission. The structural, elastic and optical properties of willemite glass-ceramics enhanced with the addition of Gd_2O_3 as dopant.

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SIFAT STRUKTUR, ELASTIK DAN OPTIK ZINK-ALUMINA-BOROSILIKA DOP GADOLINIUM OKSIDA KACA-SERAMIK

Oleh

NUR ATIKAH NAZIHAH BINTI ISMAIL

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Beberapa tahun kebelakangan ini, penghasilan dan kajian terhadap willemite (Zn_2SiO_4) berasaskan kaca-seramik telah berjaya menarik minat dan tumpuan ramai penyelidik. Walau bagaimanapun, keamatan pendarkilau wilemit berasaskan kaca-seramik didopkan dengan gadolinium oksida Gd_2O_3 kurang dilaporkan. Oleh itu, kajian ini menumpukan untuk fabrikasi dan penghasilan wilemit berasaskan kaca-seramik dengan menggunakan Gd_2O_3 sebagai bahan pengedop. Sistem kaca zink-alumina-borosilika (ZnO-Al_2O_3-B_2O_3-SiO_2) didopkan dengan Gd_2O_3 telah dihasilkan melalui kaedah sepuh lindap menggunakan komposisi $(60–x)ZnO–5Al_2O_3–15B_2O_3–20SiO_2:x(Gd_2O_3)$($x$ = 0, 0.5, 1.0, 2.0 dan 3.0 mol%) dan wilemit berasaskan kaca-seramik diperolehi dari bahan pemula kaca melalui proses penghabluran yang dikawal.

Menurut semua analisis, wilemit berasaskan kaca-seramik terbaik telah dipilih daripada sistem kaca apabila didopkan dengan 3 mol% Gd_2O_3 dan bersuhu pada 800 °C. Kaedah XRD dan FESEM digunakan untuk menganalisis sifat struktur bahan pemula kaca, penghasilan fasa hablur wilemit, bentuk dan saiz apabila suhu proses penghabluran meningkat. Purata anggaran saiz hablur halus ditentukan menggunakan XRD adalah dalam julat 50-70 nm. Sifat struktur kaca dan kaca-seramik dinilai menggunakan FTIR spektroskopi. Kajian spektrum inframerah mendedahkan kewujudan kumpulan getaran SiO_2 dan ZnO_4 menunjukkan pertumbuhan fasa hablur wilemit. Manakala untuk analisis elastik, nilai uji kajian modulus kenyal diperoleh daripada pengukuran halaju gelombang yang menggunakan teknik tak musnah ultrasonik. Halaju membujur dan ricih berubah dari 4798 ke 6976 m/s dan 2991 ke 3082 m/s masing-masing. Uji kajian modulus kenyal (modulus membujur (L), modulus ricih (G), modulus pukal (K) and modulus Young (E)) meningkat dari 94.60 ke 202.98 GPa, 36.77 ke 39.61 GPa, 45.56 ke 150.17 GPa dan 86.94 ke 109.22 GPa masing-masing. Tambahan pula, tenaga jurang jalur kaca dan kaca-seramik berkurang dari 3.64 ke 3.38 eV oleh kerana penyusunan semula struktur rangkain. Spektrum pancaran gadolinium ion menunjukkan puncak pancaran kuat di panjang gelombang 425, 447, 462, 485 dan 530 nm. Fasa wilemit menunjukkan pancaran

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utama berwarna hijau dilokasi 530 nm. Spektrum pancaran tersebut dihasilkan apabila kandungan bahan pengedop dan suhu rawatan haba meningkat, prestasi pendarkilau kaca-seramik juga bertambah baik. Penerapan gadolinium ion ke dalam hablur wilemit apabila suhu rawatan haba meningkat mempengaruhi keamatan pancaran. Sifat struktur, elastik dan optik willemite berasaskan kaca-seramik dipertingkatkan dengan penambahan Gd_2O_3 sebagai bahan pengedop.

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

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LIST OF ABBREVIATIONS

Symbols	Description
Al_2O_3	Aluminium oxide
B_2O_3	Boron trioxide
BaO	Barium oxide
CaO	Calcium oxide
DSC	Differential scanning calorimetry
E_g	Optical band gap
E	Young's modulus
FTIR	Fourier transform infrared
FESEM	Field emission scanning electron microscopy
Gd^{3+}	Gadolinium ions
Gd_2O_3	Gadolinium oxide
G	Shear modulus
JCPDS	Joint committee on powder diffraction standards
K_2O	Potassium oxide
K	Bulk modulus
L	Longitudinal modulus
MgO	Magnesium oxide
Na_2O	Sodium oxide
PLD	Pulsed laser deposition
PL	Photoluminescence
SiO_2	Silica oxide
UV	Ultraviolet
UV-Vis	Ultraviolet-Visible

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V_m	Molar volume
V_L	Longitudinal ultrasonic velocity
V_S	Shear ultrasonic velocity
XRD	X-Ray diffraction
Zn_2SiO_4	Willemite
$Zn_2SiO_4:Gd^{3+}$	Willemite doped Manganese Oxide
ZABS	Zinc-alumino-borosilicate
ZnO	Zinc oxide
α	Alpha
β	Beta
γ	Gamma
ρ	Density
σ	Poisson's ratio

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CHAPTER 1

INTRODUCTION

1.1 Research background

In recent years, researchers are developing a great interest toward transparent glass and glass-ceramics due to availability as attractive materials in optical devices. Transparent glass-ceramics have great shaping versatility of glasses and the optical efficiency of crystals (Yaowakulpattana et al., 2015). Furthermore, due to the outstanding optical properties for neon discharge lamps, fluorescent lamps, color TVs, light emitting diodes, oscilloscopes, and other displays and lighting devices, the manufacturing and analysis of glass and glass-ceramics based phosphors has been explored (Bernardo et al., 2008; Takesue et al., 2009; Sahu et al., 2016).

Transition metal and rare earth ions doped glass and glass ceramics are interesting optical host materials because they boost the optical properties of the system by exhibiting high transparency from the ultraviolet and infrared regions, a low refractive index, thermal stability, and a high likelihood of assimilating a large number of dopant ions (Hou et al., 2014). Several studies on synthesize of willemite glass-ceramics doped with europium ions and samarium ions were reported using sintering process (Tarafder et al., 2013; Tarafder et al., 2014). From the research study, it was observed the rare earth added improving the optical quality of the material as the luminescence of europium ions and samarium ions showing emission transitions from $^5D_0 \rightarrow {}^7F_4$ and $^4G_{5/2} \rightarrow {}^6H_{11/2}$ energy state. According to the result, it was discovered red emission spectra based on absorption band around 601 and 615 nm. Recently, Effendy and coworkers synthesize and studies the optical performance of europium ions doped willemite glass-ceramic (Effendy et al., 2019). From the results, they observed strong green emission peak around 557 nm with three different transition states of europium ions photon excitation. To the best of our knowledge, no reports on the luminescence performance of gadolinium ions doped willemite glass-ceramic have been published. We discovered that among numerous rare earth ions, gadolinium ions have emerged as a superior candidate for optical host materials because it emits intense green fluorescence in the visible range. Furthermore, previous work also states that the gadolinium ions doped materials attract greater interest in lighting industry for various applications (Abo-Naf et al., 2015).

Zinc silicate (Zn_2SiO_4), known colloquially as willemite, a zinc ore mineral with a phenakite structure, is one of the most common zinc ore minerals. Willemite persists in multiple polymorphs, crystalizing in various space groups (Akimoto, 1967). Willemite powder is produced using a variety of standard processes, including solid-state diffusion, pulsed laser deposition (PLD), sol-gel forced precipitation, organometallic complex route, dry reaction, spray-pyrolysis,

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combustion methods, polymer aided and hydrothermal procedures (Su et al., 1996; Bhatkar, 2007; Sharma & Bhatti, 2011; Van & Klement, 2016). However, the most preferred technique to obtain willemite is melt-quenching method because it is simple, homogeneous and easy shapes formation (Abdel-hameed & Margha, 2020). Additionally, the excellent properties of willemite, such as chemical stability, transparency in the visible range, and ultraviolet transparency, will contribute to enhance the optical characteristics that meet the criteria for phosphor materials in a variety of applications, such as televisions, fluorescent lamps, and other lighting or display devices (Brunold et al., 1996; Chakradhar et al., 2004; Wan et al., 2006; Yan & Huang, 2007; Takesue et al., 2009;).

In the proposed research, a series of gadolinium ions doped zinc-alumino-borosilicate (ZABS) glasses are made using a standard melt-quenching approach. The gadolinium ions doped willemite glass-ceramics are obtained by a controlled heat treatment procedure from these precursor glasses. Characterization of the precursor glass and willemite based glass-ceramics in terms of structural, elastic, and optical performance were conducted to evaluate the influence of heat treatment temperature and Gd_2O_3 doping. X-ray diffraction (XRD) and Fourier transform infrared (FTIR) were employed to study the structural characteristics of precursor glass and gadolinium ions doped willemite glass ceramics at varying heat treatment temperatures, while ultrasonic velocity was used to study the elastic properties. The optical properties were studied using UV-Visible (UV-Vis) and photoluminescence (PL) spectroscopy. Thus, the focus of this research is to fabricate and characterize gadolinium ions doped willemite glass-ceramics in a zinc-alumino-borosilicate (ZnO-Al_2O_3-B_2O_3-SiO_2) glass system.

1.2 Problem statement

Previously, willemite glass-ceramics materials containing transition metal and rare earth ions have been encounter greater emphasis. Rare-earth doped willemite glass-ceramics are piquing the scrutiny of scientists in laser cooling, solid-state lasers, optical devices for three-dimensional color displays, optical communications, and upconverting optical components (Tick et al., 1995; Sohn et al., 1999; Suzuki & Ohishi, 2004; Campbell et al., 2011; Nemova & Kashyap, 2012). Given the fact that willemite glass-ceramics is significant with high chemical and physical stability and high luminescence efficiency (El Ghoul et al, 2012c). Generally, willemite glass-ceramics as luminescent materials produce emission of light due to absorption of energy. Impurities and defects in crystals obtained in willemite glass-ceramics causing luminescence instability due to low energy absorption rates. In order to obtain high intensity luminescence, willemite glass-ceramics have high possibility of being a multi-color phosphor by doped with various rare earth ions producing emission ranging from ultraviolet to infrared spectral range (El Ghoul, 2018). Several reports on luminescence performance of willemite glass-ceramics doped with different rare earth ions such as manganese ions for green emission, gallium ions for violet emission, cerium ions for blue emission and europium ions for red emission color (Lu & Yun, 2013; El Ghoul, 2018; Nuraidayani et al., 2019). However, to the best of our

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knowledge, no reports on the luminescence performance of gadolinium ions doped willemite glass-ceramic have been published. We discovered that among numerous rare earth ions, gadolinium ions have emerged as a superior candidate for optical host materials because it emits intense green fluorescence in the visible range. Besides, Gd^{3+} increase energy transfer efficiency towards Ce^{3+}, Tb^{3+} and Mn^{3+} luminescent center that act as a sensitizer in scintillator or phosphor materials (Martino et al., 2008). Furthermore, previous work also states that the gadolinium ions doped materials attract greater interest in lighting industry for various applications (Abo-Naf et al., 2015).

Usually, band-gap energy of willemite glass is related with electronic band structure that depending on the temperature (Sessolo & Bolink, 2011). Energy bandgap tend to decreased with progression of temperatures leading to increase in atomic vibrations and resulting to widen average interatomic spacing (Alibe et al., 2021). Generally, low green emission color and high optical band gap of zinc silicate glass were overcome by heat treatment process to produce multiple emissions with wide optical band gap in willemite glass-ceramics. To address this issue, ZnO-Al_2O_3-B_2O_3-SiO_2 (ZABS) glass systems doped with Gd_2O_3 are synthesized followed by controlled heat treatment process to study their influence on the properties of willemite glass ceramics.

In the proposed research, a series of gadonium doped zinc aluminium borosilicate (ZABS) glasses are made using a standard melt-quenching approach. The Gd^{3+}-doped willemite glass-ceramics are obtained by a controlled heat treatment procedure from these precursor glasses. Characterization of the precursor glass and willemite based glass-ceramics in terms of structural, elastic, and optical performance were conducted to evaluate the influence of heat treatment temperature and Gd_2O_3 doping. X-ray diffraction (XRD) and Fourier transform infrared (FTIR) were employed to study the structural characteristics of precursor glass and Gd^{3+}-doped willemite glass ceramics at varying heat treatment temperatures, while ultrasonic velocity was used to study the elastic properties. The optical properties were studied using UV-Visible (UV-Vis) and photoluminescence (PL) spectroscopy. Thus, the focus of this research is to fabricate and characterize Gd^{3+}-doped willemite glass-ceramics in a ZnO-Al_2O_3-B_2O_3-SiO_2 glass system.

1.3 Objectives of the study

The primary goal of this research is to build and synthesize willemite-based glass-ceramics doped with gadolinium oxide. This project entails the identification of glass with various compositions, the melt-quenching method, a variety of heat treatment processes, the advancement of the doping process and fundamental studies of the crystallization process.

1. To synthesize the Gd^{3+}-doped willemite glass-ceramics derived from ZnO-Al_2O_3-B_2O_3-SiO_2 glass system.

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2. To study the effect Gd_2O_3 doping on structural, elastic and optical properties of precursor glass and willemite glass-ceramics.
3. To analyze the influence of various heat treatment temperatures on structural, elastic and optical properties of precursor glass, willemite based glass-ceramics and Gd^{3+}-doped willemite glass-ceramics.

1.4 Scopes of the study

The study's scopes are as follows in order to achieve the study's goal:

1) A series of precursor glass based on stoichiometric equation of (60-x)ZnO-5Al_2O_3-15B_2O_3-20SiO_2-x(Gd_2O_3) where x = 0, 0.5, 1.0, 2.0 and 3.0 mol% has been prepared using ZnO, Al_2O_3, B_2O_3, SiO_2 and Gd_2O_3 powder by simple melt-quenching technique.
2) The glass transition temperature (T_g) and glass crystallization temperature (T_c) have been measured using DSC spectroscopy.
3) Willemite based glass-ceramics has been derived from the precursor ZnO-Al_2O_3-B_2O_3-SiO_2 (ZABS) glass system by a controlled crystallization process.
4) The structural, elastic and optical properties of precursor ZABS glass, willemite glass-ceramics and Gd^{3+}-doped willemite glass ceramics has been analyzed using Archimedes method, XRD, FTIR, FESEM, ultrasonic velocity, UV-Vis and PL spectroscopy.

1.5 Hypothesis

Hypothesis of the study are related with gadolinium ions that improving the properties of network. Gadolinium ions has many strong absorption bands that emit one of strong green fluorescence and thus, improving the optical properties of glass and glass-ceramics. Therefore, incorporation of gadolinium ions in ZABS glass systems will improve the optical properties of glass and willemite glass-ceramics.

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1.6 Importance of the study

Nowadays, due to excellent luminous materials, many glass systems have a wide range of applications in a variety of sectors. To optimize the structural, elastic, and optical characteristics of transparent glass systems and glass-ceramics, transition metal (TM) and rare earth (RE) ions are doped into the systems. A vast amount of research is focusing on the production and synthesizing of glass and glass-ceramics based phosphor doped with gadolinium ions due to possible applications in lighting industry.

Willemite is a prominent and frequently used phosphor in optic, optoelectronic, and lighting technologies. All of the atoms are in the same general location and are made up of a tetrahedral framework. As a result, willemite is recognized as a good host matrix for several TM and RE ions in order to achieve high efficiency luminescence (Tarafder et al., 2014).

There have been few recent publications on the structural, elastic, and optical characteristics of Gd^{3+}-doped willemite glass-ceramics generated from precursor glass. The current study reports on the production of a precursor $ZnO-Al_2O_3-B_2O_3-SiO_2$ glass system using a traditional melt-quenching approach and the derived willemite-based glass-ceramics using a control crystallization process of precursor glasses. Thus, the willemite glass-ceramics has been doped with gadolinium oxide (Gd_2O_3) to increase the properties and quality of the final products.

1.7 Outline of thesis

The thesis consists of five chapters and the arrangement is structured as follows. Chapter 1 gives an introduction of research background that consists of willemite glass ceramics and Gd^{3+}-doped willemite glass-ceramics, the problem statements, the objectives, the scopes and also the importance of this study. Chapter 2 discussed glass theory, glass-ceramics, and earlier studies, both past and present, that have been carried out by other researchers as a guideline and citations. The list of substances, apparatus, method to prepare the samples and characterization of the precursor glass, willemite glass-ceramics and willemite glass-ceramics doped gadolinium oxide are elaborated in Chapter 3. Chapter 4 is the most significant part as the result concerning the effect of different percentage Gd_2O_3 doping content and progression of heat treatment temperatures towards the structural, elastic and optical properties of precursor glass $ZnO-Al_2O_3-B_2O_3-SiO_2$ and Gd^{3+}-doped willemite glass-ceramics are analyzed and discussed in Chapter 4. Lastly, the conclusion and future work suggestions are stated in Chapter 5.

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