UNIVERSITI PUTRA MALAYSIA

PHYSICAL, STRUCTURAL, AND OPTICAL PROPERTIES OF ZINC SILICATE-BASED GLASS-CERAMIC DERIVED FROM ZINC BORO SODA LIME SILICA GLASS SYSTEM

MUHAMMAD FARIS SYAZWAN BIN MOHD SHOFRI

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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 Master of Science

May 2021

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DEDICATION

In the name of Allah the most gracious and the most merciful

This success is dedicated to my beloved parents Mohd Shofri bin Mahmood and Fauziah binti Isa for their unlimited love

To my precious siblings and family
For their support and making my life full of happiness

To all my friends for the thick and thin we had been together

To all my lecturers and teachers for all knowledge they have given to me

Without all of them, this success would not be mine

Thank you all.

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

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May 2021

Chairman : Mohd Hafiz bin Mohd Zaid, PhD
Faculty : Science

Zinc silicate or willemite (Zn_2SiO_4) is a crystal that have unique optical characteristic which make it as a potential material in optoelectronic applications. It is also suitable for application in coating, catalyst, cathode ray tube (CRT), plasma display panels (PDPs), laser, and electroluminescent devices. In this study, a ZnO–SLS–B_2O_3 glass system have been fabricate using conventional melt-quench method and followed by an isothermal heat treatment process to synthesize zinc silicate based glass-ceramics. This project using empirical formula of $(ZnO)_{60-x}(SLS)_{40}(B_2O_3)_x$ where x = 5, 10 and 15 wt.% and heat treated at different temperature from 600 °C to 900 °C with three different holding time of 2, 4, and 10 hours. The physical, structural and optical properties were measured by using average density measurement, linear shrinkage, X-Ray diffraction (XRD), Fourier transform infrared reflection (FTIR), ultraviolet-visible spectroscopy (UV-Vis), energy band gap calculation and photoluminescence spectroscopy (PL). The density of precursor glass measurement show an increased trend from 2.726 to 2.754 g/cm^3 as ZnO content increase. The density and linear shrinkage of glass-ceramic show increased trend as progress of heat treatment temperature and duration. The XRD results show the zinc silicate crystal begins to grow after the glass sample being treated at 700 °C for 2 hours. XRD data also observed that the crystallinity of glass-ceramic increased as heat treatment increase. Besides, the FTIR spectra indicated the crystallization of zinc silicate phase occurred with the appearance of SiO_4 and ZnO tetrahedral which indicate the formation of zinc silicate crystal with the progress in heat treatment temperature and duration. In addition, UV-visible revealed slight variations where the energy of the optical band gap of precursor glass decreased from 4.40 to 4.00 eV due to the increase in ZnO content. The optical band gap of glass-ceramic also decrease with the progress in heat treatment temperature and duration. The PL spectroscopy also revealed an improvement in broad green emission at 550 nm after excite at

360nm wavelength due to the development of zinc silicate crystal in the glass-ceramic samples. From the results, with such optical properties of the zinc silicate glass-ceramic is expected to find potential application in phosphors and opto-electronic devices.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

SIFAT FIZIKAL, STRUKTUR, DAN OPTIK DARI KACA-CERAMIK BERASASKAN-ZINC SILIKA DIPEROLEHI DARI SISTEM KACA ZINK BORO SILIKA SODA LIME

Oleh

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Willemite atau zink silika (Zn_2SiO_4) adalah kristal yang mempunyai ciri optik yang unik yang menjadikannya berpotensi dalam bahan optolektronik. Ini juga sesuai untuk aplikasi pada lapisan tiub sinar katod (CRT), panel paparan plasma (PDP), alat laser, bahan pencahayaan, perangkat elektroluminecent, dan sebagai pemangkin. Dalam kajian ini, proses lindapan leburan konvensional pada kaca ZnO–SLS–B_2O_3 telah dilakukan dengan diikuti proses rawatan haba isotermal untuk menghasilkan seramik kaca zinc silicate. Projek ini menggunakan formula empirik $(ZnO)_{60-x}(SLS)_{40}(B_2O_3)_x$ dimana x = 5, 10 and 15 wt.% yang terdiri daripada komposisi 45ZnO, 50ZnO dan 55ZnO dengan lima rawatan haba berbeza iaitu 600 ℃, 700 ℃, 800 ℃ dan 900 ℃ dengan masa 2, 4, dan 10 jam. Sifat fizikal, struktur dan optik diukur dengan menggunakan ketumpatan, pengecutan linear, difraksi sinar-X (XRD), pantulan inframerah transformasi Fourier (FTIR), spektroskopi sinar ultraviolet (UV-Vis), tenaga jurang jalur dan spektroskopi fotoluminesen (PL). Pengukuran ketumpatan kaca asal zink silika menunjukkan peningkatan trend dari 2.726 hingga 2.754 g/cm^3 apabila kandungan ZnO meningkat. Manakala, pengukuran ketumpatan dan pengecutan linear kaca seramik zink silika didapati meningkat apabila terdapat peningkatan suhu rawatan haba dan masa rawatan haba. Kajian XRD menunjukkan bahawa kristal zink silika mula terbentuk setelah sampel diproses pada suhu 700 °C. Data XRD juga dapat diperhatikan bahawa terdapat peningkatan dalam pembentukkan kristal dalam kaca seramik dengan kenaikkan suhu rawatan haba. Selain itu, spektrum FTIR menunjukkan terdapat fasa spektrum zink silika dengan kemunculan SiO_4 dan ZnO_4 yang menunjukkan pembentukan zink silica dengan kenaikkan suhu dan tempoh rawatan haba. Data UV-vis menunjukkan terdapat sedikit variasi di mana tenaga jurang optik kaca pendahuluan berkurang dari 4.40 hingga 4.00 eV kerana peningkatan kandungan ZnO. Ukuran jurang jalur optik kaca seramik juga menunjukakan penurunan dengan peningkatan suhu rawatan haba dan

masa rawatan haba. Spektroskopi PL juga menunjukkan peningkatan dalam penghasilan warna hijau yang tinggi pada 550 nm setelah peningkatan suhu rawatan haba. Dengan sifat optik yang diperolehi seramik kaca ini berpotensi untuk diaplikasikan dalam fosfor dan peranti opto-elektronik.

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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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This is to confirm that:

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LIST OF ABBREVIATIONS AND SYMBOLS

Zn_2SiO_4	zinc silicate
Mn	Manganese
Eu	Europium
Co	Cobalt
ZnO	Zinc oxide
SLS	Soda lime silica glass
B_2O_3	Boron oxide
Al_2O_3	Aluminium oxide
CaO	Calcium oxide (lime)
SiO_2	Silicon dioxide
Na_2O	Sodium oxide
SO_3	Sulfur trioxide
Fe_2O_3	Ferric oxide
K_2O	Potassium oxide
BaO	Barium oxide
MgO	Magnesium oxide
MnO	Manganese oxide
XRD	X-ray diffraction
FTIR	Fourier transform infrared spectroscopy
UV-Vis	Ultraviolet-Visible
PL	Photoluminescent
α	Alpha phase
β	Beta phase
γ	Gamma phase

CHAPTER 1

INTRODUCTION

1.1 Research background

Nowadays, glass is a very versatile material that has variety of applications such as in optics as data transmission, sensor detection, technology of sensor and the optoelectronics field, as light emitting and laser diodes (Zhou et al., 2007; Hager et al., 2011; Khalkhali et al., 2012; Karaksina et al., 2013). However, there are still tons of glass waste are been thrown into the landfill every day. Glass is currently one of the least reused products in most countries and needs comparatively high quantities of energy to extract the raw materials (Poutos et al., 2008). In Malaysia, 600 tons new bottles are manufactured by glass bottles manufactures on a daily basis but just 10% of the bottle will be returned to the manufacturers and reworked to produce new bottles (Krishnamurthy and Zujip, 2013). This activity are the cause of extensive usage of landfill sites, which restricts the life cycle of landfill sites in that region, will produce not only an environmental crisis, but also an economically unsustainable one for the nation (Agamuthu et al., 2011; Moh and Manaf, 2014).

The most common glass type soda lime silica (SLS) glass that is widely used for containers, glass panes and window glass (Sheng et al., 2002). SLS glass from municipal waste consisting primarily of silicone (SiO_2), sodium (Na_2O) and calcium oxides (CaO). SLS glass are regarded as a potential candidate for the absolute or partial substitution of natural fluxes because of its low-temperature viscous flow sintering capability (Zaid et al., 2011). Therefore, there are many research have been done on recycling of glass and one of these study is on glass-ceramic. Glass with an acceptable chemical composition could be processed to glass-ceramics by controlled heat treatment method (Rawlings et al., 2006; Salman et al., 2015). Most importantly, using SLS glass will reduce the cost of production and is an effective host matrix because it has low softening point, high UV transparency, mechanical properties and good chemical durability (Attila et al., 2013; Almasri et al., 2017).

Glass-ceramic has adjustable thermal expansion coefficient, high wear resistance, low dielectric constant, high thermal stability, excellent mechanical properties, high corrosion resistance, and good thermal shock resistance (Rasteiro et al., 2007; Sarrigani et al., 2015). In addition, glass-ceramics with such a consistent crystal structure, with no porosity and a broad variety of characteristics that can be adjusted by changing the glass chemical composition (Hu et al., 2005; Yekta et al., 2007). Nowadays, research on the development and synthesize zinc silicate doped with rare earth materials and zinc silicate based glass-ceramics have produced a number of studies (Kang and Park, 2000; Xu et al., 2004; Yanbo et al., 2006; Chimalawong et al., 2012;

Tarafder et al., 2013). Zinc silicate also known by researchers as willemite is the best and most suitable host matrix for wide range of optoelectronic materials and glass phosphor, as zinc silicate has energy band gap is about 5.5 eV, high glass clarity and high chemical compound stability (Kang and Park, 2000).

Several approaches are used to fabricate zinc silicate, such as sol–gel (Babu and Buddhudu, 2013), spray pyrolysis (Kang and Park, 2000), polymer sonochemical (Masjedi and Salavati, 2016), hydrothermal (An et al., 2010) and simple thermal treatment (Su et al., 1996). However, most of these approaches are expensive because these processes is time demanding, with complicated experimental methods and limited manufacturing quantities. Moreover, the pure silica (SiO_2) sources in fabrication process is costly and used high energy due to expensive price of pure material and its high melting point (Zaid et al., 2016).

To overcome this problem another alternative source and method need to be develop in hope of lowering the cost of zinc silicate product. Therefore, in this study SLS glass is used as alternative source of silica in fabrication of zinc silicate. On the other hand, the conventional approaches such as the solid state heat treatment process has advantage of processing and synthesizing in a short period of time for experimental processing and large-scale development (Tarafder et al., 2013). Moreover, the implementation of SLS glass waste will also increase transparency, decrease the melting point, and strengthen the chemical stability, solubility and thermal stability of the finished product (Xu et al., 2004; Yanbo et al., 2006; Chimalawong et al., 2012).

Thus in this study, zinc silicate based glass-ceramic derived from the ZnO–SLS–B_2O_3 glass system was fabricated using melt-quenching method and followed by control heat treatment process. The physical characteristic of the zinc silicate based glass-ceramic has been determined using the density and linear shrinkage measurement. Besides, the structural properties of the glass and glass-ceramics were measured using X-ray diffraction (XRD) and Fourier transform infrared (FTIR). The optical properties of the glass and glass-ceramics were analysed using UV-Visible (UV-Vis) and photoluminescence (PL) spectroscopy. The results provided practical value for the utilization of waste glass for the preparation of zinc silicate based glass-ceramics, which can be used as phosphor materials in optoelectronics applications.

1.2 Problem statement

Malaysia is fast growing country the economic progress is largely depend on its manufacturing industry. This manufacturing sector continues to be the critical element for the economic and production development. However, a rapid transition in economic growth produces large volumes of waste products, such as chemicals, concrete, paper, glass and others. Malaysians were estimated to have collected 25,000 tons of garbage per day in 2012, above the projected production of 30,000 tons by 2020 (Aja and Al-kayiem, 2014). In this country, more than 90% of overall waste products have been disposed in landfills since it was the most economical disposal choice in Malaysia. One of major factors to this issue is glass waste that been generated every day by 600 tons of fresh new glass bottles and only recycled by less than 10% (Krishnamurthy and Zujip, 2013). Developments of zinc silicate using waste materials such as SLS glass as a silica source have been created to address this issue.

Furthermore, in comparison relative to many other traditional glass systems, SLS glass has attracted a lot of interest due to its superior glass forming nature. (Abbasi and Hashemi, 2014). SLS glasses often comprise of Na_2O, CaO, SiO_2, and Al_2O_3 are suitable host matrix for rare earth ions due to its high chemical stability, clarity, high thermal stability, high solubility and glass forming nature for rare earth ions (Omar et al., 2016). SLS glass may be considered a potential option for natural fluxes due to its chemical properties and low-temperature viscous flow sintering capability (Matori et al., 2010). Moreover, using SLS glass waste as alternative source of silica also help to lowering the expensive production cost of zinc silicate. This is because fabrication technique such as solid-state sintering method for industrial inorganic zinc silicate phosphors is complex and costly. This is attributed to pure starting materials (ZnO and SiO_2) which has high melting point that caused the mixture of starting material to be heated at very high temperatures for many hours (Zaid et al., 2016).

On the other hand, the B_2O_3 is added to the glass system which is a glass former that has lowers the melting point of the glass compared to SiO_2. As a result, the glasses containing these ions have a relatively low softening point and this uncommon softening feature will boost the performance of ceramic glass at lower temperatures than those usually needed for crystallization of ceramic glass material (Tarafder et al., 2013). In addition, the B_2O_3 also help to decrease the crystallization temperature and transition temperature of the glass system which help in enhance the development process of zinc silicate glass-ceramic (Sun et al., 2019). Besides, there are limited reports and systematic study of physical, structure, and optical properties of ZnO–SLS–B_2O_3 glass system and glass-ceramics produced from this glass system.

1.3 Research objective

The aim of this study is to develop and optimize the preparation of zinc silicate glass and zinc silicate glass ceramic glass systems. This project is carried out based on several goals as set out in the following:

1. To fabricate series of glass and glass-ceramic from ZnO–SLS–B_2O_3 glass system.
2. To study the effect of ZnO and B_2O_3 ratio on the physical, structure and optical properties of glass and glass-ceramics.
3. To investigate the effect of various heat treatment duration and temperatures on the physical, structure and optical properties of glass and glass-ceramics.

1.4 Scope of the study

In order to achieve the goal of the study, the focus scope of the study is as follows:

1. A series of precursor glass based on the formula $(ZnO)_{60-x}(SLS)_{40}(B_2O_3)_x$ where x = 5, 10 and 15 wt.% has been prepared using SLS glass, ZnO and B_2O_3 powder by conventional melt-quenching technique.
2. The composition and the structural of the precursor ZnO–SLS–B_2O_3 glass system has been measured using XRD and FTIR in order to confirm the amorphous structural of glass and chemical bonding in the glasses.
3. Zinc silicate based glass-ceramics has been developed from the precursor ZnO–SLS–B_2O_3 glass system by a controlled crystallization process using conventional heat treatment method.
4. The physical, structural and optical characteristic of zinc silicate glass-ceramics base ZnO–SLS–B_2O_3 glass has been analysed using Archimedes method, linear shrinkage, XRD, FTIR, UV-Vis and PL spectroscopy.

1.5 Important of the study

Today, many countries are having a difficulty to dispose solid wastes from industries. These countries have small disposal areas to dispose their waste products. Recycling of waste materials such SLS glass was reported by other researchers to reduce solids wastes. In addition, glass and glass-ceramics from waste materials such as zinc silicate have gained from the attention of the researcher and have been extensively researched. This is due to the fact that zinc silicate is suitable material as a phosphor in many applications due to its phenakite structure and due to its rigid lattice with non-centrosymmetric cationic sites which helps to improve its optical properties (Sarrigani et al., 2015). This thesis centred on the phase transformation of glass-ceramics derive from ZnO–SLS–B_2O_3 by using waste materials. In this research, the preparation ZnO–SLS–B_2O_3 of precursor glass system by melt-quenching method and produce zinc silicate glass-ceramics using control sintering process of glasses is reported.

1.6 Outline of Thesis

The thesis is structured as follows. Chapter 1 points out the concept of zinc silicate base glass-ceramic, the problem statements, the objectives, the scope and also the significance of this research. Previous study, both past and present that has been carried out by other global researchers was covered throughout the literature review in chapter 2. The methods used to prepare zinc silicate and the characterization used is stated in Chapter 3. Analysis of the effect of composition and heat treatment temperatures on the physical, structural and optical properties of precursor glass and zinc silicate ceramic are examined and presented in Chapter 4. Finally, Chapter 5 offers a conclusion and a recommendation for potential future work.

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