UNIVERSITI PUTRA MALAYSIA

PHYSICAL, STRUCTURAL AND OPTICAL PROPERTIES OF WILLEMITE-BASED GLASS-CERAMIC DOPED WITH MANGANESE OXIDE

MOHD HAFIZ BIN MOHD ZAID

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By

MOHD HAFIZ BIN MOHD ZAID

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

May 2016

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Dedicated to

To my beloved parents Mohd Zaid bin A. Bakar and Kamsiah binti Mustaffa
For their unconditional love and support

To my siblings and family
For making my life complete

To my beautiful wife Nurzia binti Mohamad and her family
For their love and care

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

To all my lecturers
For helping me at a lot throughout this journey

Thank you all

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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May 2016

Chairman: Assoc. Prof. Khamirul Amin bin Matori, PhD
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Over the last few decades, great deals of interest have been focused on the fabrication and synthesizing willemite based ceramic. However, only few of them used waste materials to fabricate the willemite in form of glass-ceramic. This research was carried out based on several objectives such as to synthesis and study the effect of ZnO addition on the physical, structure and optical properties of soda lime silica (SLS) glass system. Besides, this work also investigation the effect of various heat treatment temperatures on the physical, structure and optical of precursor glass and glass-ceramics and the influence of MnO doping on the physical, structure and optical properties of willemite based glass-ceramics. Thus, in this study, willemite based glass-ceramics were fabricate and synthesized using SLS glass waste as a source of silicon. A series of precursor glasses in the ZnO-SLS glass system was prepared by the conventional melt-quench technique. Willemite based glass-ceramics were derived from the selected precursor ZnO-SLS glass by a controlled crystallization process. The thermal, chemical and physical properties of precursor glass and glass-ceramics were measured by differential thermal analysis (DTA), energy dispersive X-ray fluorescence (EDXRF), average density and linear shrinkage measurement. The average density and linear shrinkage of glass and glass-ceramic samples were found increased with increasing of heat treatment temperature and concentration of dopant. Besides, the structural properties of precursor glass and formation of willemite crystal phase, morphology and size with increase in heat-treatment temperatures was examined by X-ray diffraction (XRD) and field-emission scanning electron microscopy (FESEM) techniques. The average calculated crystallite size obtained from XRD was found to be in the range 30-60 nm whereas the grain size observed in FESEM was 300-700 nm. Fourier transform infrared (FTIR) reflection spectroscopy was used to evaluate the structural of glass and glass-ceramics. The appearance of SiO_2, ZnO_4 and Zn-O-Si bands detected from FTIR measurements indicate that the formation of willemite crystal phase. The study of the optical band gap has found that optical band gap of glass and glass-ceramics decreased as the percentage of dopant and heat treatment temperature increased. The

photoluminescence spectra of Mn^{2+} ions exhibit emission transitions of $^4T_1(G)$ - $^6A_1(S)$ and its excitation spectra show an intense absorption band at 260 nm. Prominent green emission colors of α-Zn_2SiO_4 phase were observed centered at 525 nm while the yellow emission centered at 585 nm resulted from β-Zn_2SiO_4 phase. These spectra reveal that the luminescence performance of the glass-ceramics is increase with the progression of dopant percentage and heat treatment temperatures. This enhancement is caused by partitioning of Mn^{2+} ions into the willemite crystals with progress of heat treatment temperatures. Such luminescent glass-ceramics are expected to find potential applications in phosphors and opto-electronic devices.

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

SIFAT FIZIKAL, STRUKTUR DAN OPTIKAL WILLEMITE-ASAS KACA-SERAMIK DOP BERSAMA MANGAN OKSIDA

Oleh

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Sejak beberapa dekad yang lalu, penghasilan dan kajian terhadap willemite berasaskan seramik telah berjaya menarik minat dan tumpuan ramai penyelidik. Walau bagaimanapun, penyelidikan untuk menghasilkan willemite dalam bentuk kaca-seramik dengan menggunakan bahan asas dari bahan-bahan buangan adalah sangat sedikit dan terhad. Kajian ini dijalankan berdasarkan beberapa objektif seperti untuk sintesis dan mengkaji kesan penambahan ZnO pada fizikal, struktur dan ciri-ciri optik sistem kaca SLS. Selain itu, kerja-kerja ini juga penyiasatan kesan pelbagai suhu rawatan haba ke atas fizikal, struktur dan optik kaca pelopor dan kaca seramik dan pengaruh MnO dopan pada fizikal, struktur dan sifat optik willemite berasaskan kaca seramik. Oleh yang demikian, dalam kajian ini, willemite berasaskan kaca-seramik telah dihasilkan dengan menggunakan sisa kaca soda lime silika (SLS) sebagai sumber silikon dan beberapa kajian terhadap sifat-sifat willemite dalam bentuk kaca-seramik telah dijalankan. Beberapa siri kaca berasaskan sistem ZnO-SLS telah dihasilkan dengan menggunakan teknik teknik lindapan leburan. Willemite berasaskan kaca-seramik telah berjaya dihasilkan daripada kaca yang dipilih daripada siri kaca ZnO-SLS melalui proses penghabluran yang dikawal. Sifat haba, kimia dan fizikal kaca dan kaca-seramik telah diukur mengunakan alat DTA, EDXRF, purata ketumpatan dan pengukuran pengecutan linear. Didapati , purata ketumpatan dan pengecutan linear kaca dan kaca-seramik telah meningkat disebabkan oleh peningkatan suhu rawatan haba dan kepekatan dopan. Selain itu, sifat-sifat struktur kaca dan pembentukan fasa, morfologi dan saiz kristal willemite dengan peningkatan suhu rawatan haba telah diperiksa dengan menggunakan alat XRD dan FESEM. Didapati, purata saiz kristal yang diperolehi daripada XRD adalah dalam julat 30-60 nm manakala saiz butiran yang diperhatikan menggunakan FESEM adalah dalam julat 300-700 nm. FTIR spektroskopi telah digunakan untuk menilai struktur kaca dan kaca-seramik. Kemunculan dan penghasilan ikatan SiO_2, ZnO_4 dan Zn-O-Si yang dikesan dari alat FTIR telah menunjukkan bahawa pembentukan kristal willemite telah berlaku. Kajian ke atas jurang jalur optik telah mendapati bahawa jumlah jurang jalur optik kaca dan kaca-seramik telah menurun disebabkan peningkatan peratusan dopan dan suhu rawatan haba. Selain itu,

spektrum kefotopendarcahayaan daripada peralihan Mn^{2+} ion daripada $^4T_1(G)$ - $^6A_1(S)$ dan spektrum pengujaan yang menunjukkan satu jalur penyerapan pada jarak 260 nm. Warna hijau yang terhasil daripada fasa α-Zn_2SiO_4 diperhatikan berpusat pada 525 nm manakala warna kuning berpusat pada 585 nm yang terhasil daripada fasa β-Zn_2SiO_4. Spektrum ini telah mendedahkan bahawa prestasi kependarkilauan daripada kaca-seramik telah meningkat disebabkan peningkatan dalam peratusan dopan dan suhu rawatan haba. Peningkatan ini adalah disebabkan oleh pembahagian ion Mn^{2+} ke dalam kristal willemite dengan peningkatan suhu rawatan haba. Penghasilan kaca-seramik ini dijangka berpotensi untuk diaplikasikan ke dalam bahan 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 Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

Zn_2SiO_4	Willemite
$Zn_2SiO_4:Mn^{2+}$	Willemite doped Manganese Oxide
Mn	Manganese
Eu	Europium
Co	Cobalt
Ni	Nickel
UV	Ultraviolet
SLS	Soda lime silica
SiO_2	Silica oxide
ZnO	Zinc oxide
MnO	Manganese oxide
CaO	Calcium oxide
Na_2O	Sodium oxide
Al_2O_3	Aluminium oxide
K_2O	Potassium oxide
MgO	Magnesium oxide
Fe_2O_3	Ferric oxide
B_2O_3	Boron trioxide
BaO	Barium oxide
$MnCO_3$	Manganese carbonate
$ZnCl_2$	Zinc chloride
Na_2SiO_3	Sodium silicate
MnC_2O_4	Manganese oxalate
$MnCl_2$	Manganese (II) chloride
α	Alpha phase
β	Beta phase
γ	Gamma phase
PVA	Polyvinyl alcohol
EDXRF	Energy dispersive X-ray fluorescence
XRD	X-Ray diffraction
FTIR	Fourier transform infrared
FESEM	Field emission scanning electron microscopy
UV-Vis	Ultraviolet-Visible
PL	Photoluminescence
E_{opt}	Optical band gap
JCPDS	Joint committee on powder diffraction standards
NBO	Non-bridging oxygen

CHAPTER 1

INTRODUCTION

1.1 Research background

Manufacturing industry has played an important role for economic progress for the past decades. This industrial continues to maintain as the main sector for the development and economic growth. However, the fast change in industrial development generates enormous amount of waste disposal such as plastic, steel, paper, glass and others. This signaled the essential for a method to solve the waste problem through the solid waste management.

The existing of solid waste management system in Malaysia for municipal and industrial wastes gives a priority and promotes the consumption of recycle, treatment and disposal method such as composting, sanitary landfill or incineration (Victor and Agamuthu, 2013; Moh and Manaf, 2014). Nevertheless, this approach has been found creating many environmental problems such as illegal dumping, leaving large quantities of residues waste and the necessity for new land for the establishment of disposal facilities (Kadir *et al*., 2013; Teo *et al*., 2014; Zen *et al*., 2014).

The vitrification process has been known as one of promising method to solve the solid waste problems and it has been applied for the stabilization of a variety of municipal, industrial, commercial and construction waste (Kavouras *et al*., 2003). Besides, the vitrification process commonly results in a reduction of solid waste in large volume; benefits in terms of storage land or dumping site. Vitrification process is considered as an important technology for the treatment and remediation of non-combustible waste for the disposal of solid waste (Ponsot *et al*., 2015).

Recently, upcycling the solid waste into more valuable and environment friendly products is a new focus across the world. This situation is comparable with the increasing demand especially for limited natural resource. Glass is an interesting material due to its various applications and widely used in optics such as data transmission, sensor detection, technology of sensor, and it is believed the best material for solid-state lasers (Prakash *et al*., 2001; Ehrt, 2004; Zhou *et al*., 2007; Hager *et al*., 2011; Karaksina *et al*., 2013).

Glass containing zinc oxide (ZnO) is one of the most preferred glass materials due to their good mechanical properties and low softening point compared with other glass systems (Yousef *et al*., 2007; Abo-Mosallam *et al*., 2010). This type of glass is a promising optical host material in solid-state laser when doped with transition metal and rare earth ions because this glass exhibits high transparency from UV to IR region, low refractive index and thermal expansion coefficient with high thermal stability and possibility of incorporating large number of dopant ions (Rosmawati *et al*., 2008; Yu *et al*., 2008; Kim *et al*., 2009; Hou *et al*., 2014).

The conversion of waste glasses into glass-ceramics has gradually become an important method to improve the recycling of solid wastes into value-added materials (Boccaccini *et al*., 1997). Glass with a suitable chemical composition can be transformed into glass-ceramics through the controlled heat treatment process (Barbieri *et al*., 2000; Rawlings *et al*., 2006; Salman *et al*., 2015). Soda lime silica (SLS) glass is the most prevalent type of glasses and commonly used for window glass panes and glass containers (Clark *et al*., 1976; Sehgal and Ito, 1998; Sheng *et al*., 2002). SLS glass typically soften around 700 °C and this unique softening behaviour can influence the fabrication of glass-ceramic at temperatures lower than usually required to crystallize the glass-ceramic products (Zanotto, 1991; Pontikes *et al*., 2007; Petrescu *et al*., 2012; Marinoni *et al*., 2013). Hence, the parent glass is transformed into a glass-ceramic in which the crystalline phase is bonded by the residual glass. Apart from that, glass-ceramics with a uniform fine-grain microstructure, non-porosity and wide range of properties can be tailored by altering the chemical composition (Karamanov *et al*., 1999; El-Shennawi *et al*., 2001; Hu *et al*., 2005; Yekta *et al*., 2007).

Zinc silicate (Zn_2SiO_4) or also known by its mineral name willemite, is one of the zinc ore minerals with a phenakite structure. In the willemite structure, all the atoms occupy the overall position and composed of tetrahedral framework. With this kind of rigid lattice (non-centrosymmetric cationic sites), willemite provides special optical properties (Tarafder *et al*., 2014). For this reason, willemite is very important and widely used as a phosphor in optic, opto-electronic and lighting devices (Xu *et al*., 2010; Sivakumar *et al*., 2012).

In addition, willemite is known as a suitable host matrix for numerous transition metal and rare-earth ions for high efficient luminescence (Takesue *et al*., 2009; Choo *et al*., 2010; Omri *et al*., 2013). According to Takesue *et al*. (2009), the luminescent performance of the manganese doped willemite ($Zn_2SiO_4:Mn^{2+}$) phosphors has been characterized by the transition of $3d^5$ electron in the Mn^{2+} ion that acting as an activating center in the willemite structure. To be detailed, the transition from the lowest excited state, $^4T_1(G)$ to the ground state $^6A_1(S)$ transition is directly being responsible for the strong green emission under an ultraviolet light (Patra *et al*., 2005; Hao and Wang, 2007; Lukic *et al*., 2008; Seo *et al*., 2009; Diao and Yang, 2010; Uegaito *et al*., 2012; Park *et al*., 2015).

In the current study, a series of zinc soda lime silica (ZnO-SLS) glasses are prepared from conventional melt-quenching technique and willemite based glass-ceramics are derived from these precursor glasses by a controlled heat treatment process. The properties of the precursor glass and willemite glass-ceramics have been characterized such as thermal, chemical, physical, structural, morphological and optical behaviour in order to study the effect of heat treatment temperature and MnO doping. The structural and optical properties of precursor glass and Mn-doped willemite glass-ceramics have been studied by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), Fourier transform infrared (FTIR), Uv-Visible (Uv-Vis) and photoluminescence (PL) spectroscopy. In view of above fact, the aim of this work is the fabrication and characterization of Mn-doped willemite glass-ceramics in ZnO-SLS glass system as a potential materials use in the field of opto-electronic as phosphor materials.

1.2 Problem statement

In recent years, glass and glass-ceramics doped with transition metal and rare earth ions are attracting prodigious interest in solid state lasers, phosphors, optical and lighting devices (Babu *et al*., 2015). At present, researchers are aiming of using waste as starting material for producing willemite phosphor so that low energy processes and new technique may be developed. The conventional solid-state methods for commercial inorganic phosphors are completely established in industry. In this technique; pure raw materials such as ZnO and SiO_2 are well-mixed and fired at very high temperatures for several hours due to the high melting temperature of starting materials. To overcome high energy use problem, the productions of willemite using waste material such as glass waste as a silicon source are developed. Besides, there are limited reports and systematic study of physical, structure, and optical properties of precursor ZnO-SLS glass system and willemite based glass-ceramics.

Compared to several other conventional glass systems, SLS glass has drawn a great attention because of their superior glass forming nature (Abbasi and Hashemi, 2014). Furthermore, SLS glass also intriguing many researchers because of its excellent chemical, optical and mechanical properties such as low thermal expansion coefficient, nonlinear refractive index, fine chemical stability, high UV transparency, large tensile fracture strength and good durability for opto-electronic applications (Abo-Mosallam *et al*., 2010; Chimalawong *et al*., 2010; Marinoni *et al*., 2013).

The transformation of glass into glass-ceramics has progressively become an important technique to improve the quality and properties of the final products. ZnO-SLS glasses are one of the most preferred parent glasses due to their improved mechanical properties and low softening temperature point compared with other glass systems (Shelby, 2005). This type of glass transformed into glass-ceramic with willemite as a main crystal phase. Willemite has a promising future in advanced materials as a favorably flexible luminescent material due to the broad range of multi-colors that can be obtained from various transition metal ions. $Zn_2SiO_4:Mn^{2+}$ has been used as a phosphor in fluorescent lamps, neon discharge lamps, oscilloscopes, black-and-white televisions, color televisions, and many other displays and lighting devices.

For that reasons, a comprehensive study of the crystallization, properties and effect of heat treatment on Mn-doped willemite glass-ceramics derived from precursor

ZnO-SLS glass system are carried out and the results of this research are expected to find potential application as phosphor material for opto-electronic devices.

1.3 Research objective

The main objective of this project is to develop and optimize willemite based glass-ceramics derived from ZnO-SLS glasses. This work involved design of suitable glass compositions, melt-quenching, development of heat treatment, progress of doping process and a series of fundamental studies of the crystallization process.

This research was carried out based on several objectives as in the following:

1) To synthesis and study the effect of ZnO addition on the physical, structure and optical properties of SLS glass system.
2) To investigate the effect of various heat treatment temperatures on the physical, structure and optical of precursor glass and glass-ceramics.
3) To analyze the influence of MnO doping on the physical, structure and optical properties of willemite based glass-ceramics.

1.4 Scope of the study

In order to achieve the objective of the study, the scopes of the study as follow:

1) A series of precursor glass based on the stoichiometric equation of x(ZnO)100-x(SLS) where x = 0, 10, 20, 30, 40 and 50 wt.% has been prepared using SLS glass powder and ZnO powder by conventional melt-quenching technique.
2) The chemical composition of precursor ZnO-SLS glass system has been measured using EDXRF spectroscopy in order to confirm the oxide and percentage of chemical oxide in the glasses.
3) The glass transition temperature (T_g) and glass crystallization temperature (T_c) has been measured using DTA spectroscopy.
4) Willemite based glass-ceramics has been derived from the precursor ZnO-SLS glass system by a controlled crystallization process.
5) The physical, structural and optical properties of ZnO-SLS glass, willemite and Mn-doped willemite based glass-ceramics has been analyzed using Archimedes method, linear shrinkage, XRD, FESEM, FTIR, UV-Vis and PL spectroscopy.

1.5 Important of the study

Recently, glass and glass-ceramic from waste material opens a wide range of possibilities for designing new materials and attracting a significant interest in solid state lasers, phosphors and optics. A great deal of research has been focused on the fabrication and characterization of glass and glass-ceramics based phosphors in order to obtain efficient luminescent materials for fluorescent lamps, neon discharge lamps, oscilloscopes, color televisions, light emitting diode and many other displays and lighting devices (Bernardo *et al*., 2008; Takesue *et al*., 2009; Ding *et al*., 2015).

Willemite has been identified as a suitable host matrix for many transition metal dopant ions for efficient luminescence. In willemite, all the atoms occupy overall position and composed of tetrahedral framework where in zinc and silicon positioned in three different fourfold crystallographic sites. This kind of rigid lattice gives the chance to obtain better optical properties (Tarafder *et al*., 2014).

To the best of our knowledge, there are very few reports on physical, structural and optical properties of Mn-doped willemite glass-ceramics derived from precursor glass. In the present research, the preparation of precursor ZnO-SLS glass system by conventional melt-quenching technique and derived willemite based glass-ceramics by control crystallization process of precursor glasses is reported. Subsequently, the willemite glass-ceramics has been doped with manganese oxide (MnO) to increase the properties and quality of the final products.

1.6 Outline of thesis

The thesis arrangement is structured as follows. Chapter 1 gives an introduction of precursor ZnO-SLS glass, willemite glass-ceramic and Mn-doped willemite glass ceramics, the problem statements, the objectives, the scopes and also the importance of this study. The theory of glass, glass-ceramic and previous works including past and current has been carried out by other researchers are covered in Chapter 2. The apparatus, methodology and characterization of the precursor glass and willemite glass-ceramic doped manganese are explained in Chapter 3. The results concerning the effect of ZnO addition to the SLS glass, progression of heat treatment temperatures and different percentage of MnO doping content towards the physical, structural and optical properties of precursor ZnO-SLS glass and Mn-doped willemite glass-ceramic are analyzed and discussed in Chapter 4. Finally, the conclusion and suggestion for future works are presented in Chapter 5.

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