UNIVERSITI PUTRA MALAYSIA

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

NURAIDAYANI BINTI EFFENDY

FS 2017 24

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By

NURAIDAYANI BINTI EFFENDY

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

March 2017

DEDICATION

To my beloved parents Effendy Bin Harun and Norina Binti Yassin

For their unconditional love and support

To my siblings and family

For making my life complete

To all my 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 fulfillment of the requirement for the degree of Master of Science

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Chairman: Zaidan Bin Abdul Wahab, PhD
Faculty: Science

Over the past few decades, willemite based glass-ceramics doped with rare earth ions have attracted significant attention on the fabrication and characterization. However, only few of them used the waste glass materials to fabricate the willemite in the form of glass-ceramics. Hence, this study is focused on the production and characterization of willemite glass-ceramics from the SLS glass waste as a source of silicon. A series of ZnO-SLS-Er_2O_3 glass samples was prepared by the conventional melt-quench technique. Willemite based glass-ceramics were derived from controlled the crystallization process of ZnO-SLS-Er_2O_3 glass systems. The thermal, chemical and physical properties of glass and glass-ceramics samples were measured by differential thermal (DTA), energy dispersive X-ray fluorescence (EDXRF), average density and linear shrinkage measurement. The differential thermal analysis reveals that the glass transition (T_g) and crystallization temperature (T_c) decrease with the increase of Er_2O_3 content in the ZnO-SLS glass system. This decrement is due to the ability of Er_2O_3 in reducing the melt viscosity of the glass network. The elements of ZnO, SiO_2, CaO, Na_2O, Al_2O_3, Fe_2O_3, K_2O, NiO and Er_2O_3 were detected in glass samples using EDXRF measurement. The average density and linear shrinkage of glass and glass-ceramics samples are increases with the increment of sintering temperature as well as dopant concentration. Moreover, the structural properties of glass and the formation of willemite crystal phase and morphology varies with sintering temperature was studied by X-ray diffraction (XRD) and field emission scanning electron microscopy (FESEM) technique. The XRD measurement showed the formation of stable α-willemite at 700°C sintering temperature**.** Fourier transform infrared reflection (FTIR) spectroscopy is used to get more information about the functional groups of the studied glass and glass-ceramics samples. The presence of vibrations associated with the SiO_4 and ZnO_4 groups would clearly suggest the formation of Zn_2SiO_4 phase. In this study, the optical band gap of glass and glass-ceramics are found to be decrease as the sintering temperature and percentage of dopant increase. Broad green emission at 559 nm under excitation 385 nm that associated to the $^4S_{3/2} \rightarrow {}^4I_{15/2}$ transitions was obtained. These spectra reveal that the luminescence of the samples is increased with the progression of sintering temperature due to the presence of Er^{3+} ions into the willemite crystal. Such luminescence of glass and glass-ceramics are expected to have potential applications in phosphor and optoelectronic devices.

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

SIFAT FIZIKAL, STRUKTUR DAN OPTIKAL WILLEMITE BERASASKAN KACA-SERAMIK DOP DENGAN ERBIUM OKSIDA

Oleh

NURAIDAYANI BINTI EFFENDY

Mac 2017

Pengerusi: Zaidan Bin Abdul Wahab, PhD
Fakulti: Sains

Sejak beberapa dekad yang lalu, penghasilan dan pencirian willemite berasaskan kaca-seramik yang didopkan dengan ion nadir bumi telah menarik perhatian dan tumpuan ramai penyelidik. Walau bagaimanapun, penyelidikan terhadap penghasilan willemite berasaskan kaca-seramik dengan menggunakan bahan asas dari bahan-bahan buangan adalah sangat sedikit dan terhad. Justeru itu, kajian ini memberi tumpuan kepada penghasilan dan pencirian willemite berasaskan kaca-seramik dengan menggunakan sisa kaca SLS sebagai sumber silicon. Beberapa siri kaca berasaskan sistem ZnO-SLS-Er_2O_3 telah dihasilkan dengan menggunakan teknik lindapan leburan. Willemite berasaskan kaca-seramik telah berjaya dihasilkan melalui proses penghabluran yang dikawal sistem kaca ZnO-SLS-Er_2O_3. Sifat-sifat haba, kimia dan fizikal kaca dan kaca-seramik telah berjaya diukur dengan menggunakn alat DTA, EDXRF, purata ketumpatan dan pengukuran pengecutan linear. Analisis DTA menunjukkan suhu peralihan kaca and suhu penghabluran menurun dengan kenaikan kandungan Er_2O_3 di dalam sistem kaca ZnO-SLS-Er_2O_3. Penurunan ini adalah disebabkan oleh keupayaan Er_2O_3 dalam mengurangkan kepekatan rangkaian kaca. Unsur ZnO, SiO_2, CaO, Na_2O, Al_2O_3, Fe_2O_3, K_2O, NiO dan Er_2O_3 telah dikesan di dalam sampel kaca dengan menggunakan pengukuran EDXRF. Dalam kajian ini, didapati bahawa purata ketumpatan dan pengecutan linear untuk kaca dan kaca-seramik telah meningkat selaras dengan peningkatan suhu pensinteran dan juga kepekatan dopan. Selain itu, sifat-sifat struktur kaca dan pembentukan morfologi dan fasa kristal willemite dengan peningkatan suhu pensinteran telah dikaji dengan menggunakan kaedah XRD dan FESEM. Pengukuran XRD menunjukkan pembentukkan α-willemite dapat dikesan pada suhu pensinteran 700 °C. FTIR spektroskopi pula digunakan untuk mendapatkan maklumat yang lebih lanjut mengenai fungsi kumpulan atau mengenal pasti sebatian di dalam kaca dan kaca-seramik sampel. Dengan kehadiran dan penghasilan ikatan SiO_4 dan ZnO_4 yang dikesan daripada alat FTIR, jelas menunjukkan bahawa pembentukan kristal willemite telah berlaku. Kajian ini juga mendapati bahawa jurang jalur optik kaca dan kaca-seramik telah menurun disebabkan oleh peningkatan peratusan dopan dan suhu pensinteran. Spektrum berwarna hijau berpusat pada 559 nm telah terhasil di bawah pengujaan satu jalur penyerapan pada jarak 385 nm yang dikaitkan kepada peralihan $^4S_{3/2} \rightarrow {}^4I_{15/2}$ telah diperolehi. Spektrum ini telah menunjukkan bahawa prestasi kependarkilauan sampel telah meningkat dengan peningkatan suhu pensinteran yang disebabkan oleh kehadiran

ion Er^{3+} ke dalam kristal willemite. Kependarkilauan oleh kaca dan kaca-seramik ini dijangka berpotensi untuk diaplikasikan ke dalam bahan fosfor dan peranti opto-elektronik.

ACKNOWLEDGEMENTS

In the name of Allah S. W.T, the Most Gracious and Most Merciful. I am very grateful to Allah for the blessing to be able to complete my thesis.

Special appreciation goes to my supervisor, Assoc. Prof. Dr. Zaidan Bin Abdul Wahab, for his supervision and constant support. His invaluable help of constructive comments and suggestion throughout the experiment and thesis works have contributed to the success of this research.

Not forgotten, my appreciation to the committee members Assoc. Prof. Dr. Halimah Binti Mohamed Kamari for the support and guidance regarding to this topic. Humble appreciation also goes to all the staff and technicians of the Faculty of Science and Institute of Advance Technology, Universiti Putra Malaysia for the co-operation and technical support provided.

Sincere thanks to all members of Glass, Ceramic and Composite Metal (GCCM) Laboratory for their helps and moral supports upon the completion of my project.

Last but not least, my deepest gratitude goes to my parents, Mr. Effendy Bin Harun and Mrs. Norina Binti Yassin for their endless love, prayers and encouragement.

May Allah bless all of the people for giving me the utmost support in completion of this research.

I certify that a Thesis Examination Committee has met on 3 March 2017 to conduct the final examination of Nuraidayani binti Effendy on her thesis entitled "Physical, Structural and Optical Properties of Willemite-Based Glass-Ceramic Doped with Erbium Oxide" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

Members of the Thesis Examination Committee were as follows:

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Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Sidek bin Hj Ab Aziz, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

Rosli Hussin, PhD
Professor
Universiti Teknologi Malaysia
Malaysia
(External Examiner)

NOR AINI AB. SHUKOR, PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 2 June 2017

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

Zaidan Bin Abdul Wahab, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Halimah Binti Mohamed Kamari, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Member)

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Signature: _____
Name of Chairman of Supervisor Committee: Zaidan Bin Abdul Wahab

Signature: _____
Name of Member of Supervisor Committee: Halimah Binti Mohamed Kamari

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LIST OF ABBREVIATIONS

Zn_2SiO_4	Willemite
Er_2O_3	Erbium Oxide
SLS	Soda lime silica
SiO_2	Silica oxide
ZnO	Zinc Oxide
Na_2O	Sodium Oxide
CaO	Calcium Oxide
Al_2O_3	Aluminium Oxide
K_2O	Potassium Oxide
MgO	Magnesium Oxide
Nd_2O_3	Neodymium Oxide
Dy_2O_3	Dysprosium Oxide
As_2O_3	Arsenic Trioxide
B_2O_3	Boron Trioxide
Fe_2O_3	Ferric Oxide
BaO	Barium Oxide
α	Alpha
β	Beta
γ	Gamma
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

CHAPTER 1

INTRODUCTION

1.1 Research background

Recently, glass-ceramics doped with rare earth ions have attracted significant attention because of their wide application in the field of laser technology and optical communication. Glass-ceramics open a great variety of potentials for designing new materials for optoelectronic industries. Generally, glass-ceramics can be formed by melting the specific glass composition and converting it into a glass-ceramic by an appropriate controlled heat treatment process. The glass-ceramics properties are mainly determined by the constituent phase and microstructure, which depend on the composition of the precursor glass (Omar and Abdel-Hameed, 2009). Besides, the heat treatment and the addition of nucleating agents also are the main point in crystal growth of glass-ceramics (Omar and Abdel-Hameed, 2009).

Indeed, various glass-ceramics such as silicates and phosphates have been developed as the proper host for rare earths. However, phosphates have been restricted used as the host for these ions due to the poor in chemical stability and low transition temperature (Capek *et al.,* 2004). In contrast, silicates show much superior chemical stability compared with phosphate and therefore it has advantages for ion-exchange to produce optical waveguides (Capek *et al.,* 2004). In addition, silicate materials are most economic due to the traditional technology used for telecommunication application.

Lately, the fabrication and characterization of a wide variety of oxide based phosphors have been a great of interest focused in order to obtain efficient luminescent materials for flat panel display device (Wu *et al.,* 2005; Takesue *et al.,* 2009; Van Der Kolk *et al.,* 2000). Among the silicate based glass-ceramics, willemite or zinc silicate (Zn_2SiO_4) has been identified as an ideal host matrix for many rare earth and transition metal dopant ions for efficient luminescence and also as materials for electronic application (Ahmadi *et al.,* 1999; Chakradhar *et al.,* 2004; Wan *et al.,* 2006). Willemite also is very important and widely used as a phosphor in neon discharge lamps, fluorescent lamps, oscilloscope, colour televisions and many other displays and lighting devices (Yan and Huang, 2007; Brunold *et al.,* 1996; Yi *et al.,* 2014; Chen *et al.,* 2010; Hu and Qiu, 2015; Miguel *et al.,* 2014; Balda *et al.,* 2011).

In advance, glass-ceramics doped with rare earth oxide give the great potential applications in different areas such as solid-state lasers, optical data storage, optical devices for the three dimensional colour displays and upconverting optical devices (Boccaccini *et al.,* 1996; Lu *et al.,* 2011; Li *et al.,* 2002). Among various rare earth ions, Er^{3+} ions is one of the importance active ions for luminescence because it can offer not only infrared luminescence for optical amplification due to their ability to increase the signal at the wavelength of 1530 nm which corresponds to the intra-4f transition of Er^{3+} from $^4I_{13/2}$ to $^4I_{15/2}$, but also one of the favourable energy level structures in near infrared

spectral region with two transitions: $^4I_{15/2} \rightarrow {}^4I_{9/2}$ (800nm) and $^4I_{15/2} \rightarrow {}^4I_{11/2}$ (980 nm) which can be efficiently excited, thus yielding blue, green and red upconversion emission (Zhang *et al.,* 2000; Shaim *et al.,* 2002).

1.2 Problem statement

In Malaysia, the amount of disposal solid waste materials increases yearly. Most of the waste is disposed in landfill sites and Malaysia has limited suitable landfill sites. Among the solid waste materials, glass is one of the disposal solid waste materials. However, glass is the material that can be recycled and reprocessed to produce new products.

In this study, soda lime silicates (SLS) glass is used as a source of silicate (SiO_2) in order to form a willemite glass-ceramic sample. However, the making of the willemite based glass-ceramics from pure silicate material was expensive. Thus, the used of SLS waste glass as a source of silicate can bring new development to the industry and consequently help the environment through waste to wealth concept.

In addition, many researches have been studying on producing willemite based glass ceramics doped with rare earth and transition metals by using pure materials. There is a limited report regarding the doping of erbium oxide into willemite, which is produced from the waste material. Therefore, a detailed investigation of the structural, chemical, morphological and optical properties for the production of willemite based glass ceramic from waste material doped with erbium oxide are presented in this the thesis.

1.3 Research objectives

This study was carried out based on a clear and precise objective. The purposes of the research are:

1. To determine the influence of various concentration of erbium oxide on the chemical composition, thermal properties, physical properties, structure features and optical properties of willemite glass-ceramics.
2. To determine the influences of varying sintering temperatures of various concentration of erbium oxide on the physical properties, structure features and optical properties of willemite glass-ceramics.

1.4 Scope of study

In order to achieve the objective of the study, the scope of the study are follows:

1. The willemite based glass-ceramic doped with Er_2O_3 samples based on the composition (in wt. %) of $[(ZnO)_{0.5}(SLS)_{0.5}]_{1-x}(Er_2O_3)_x$ where x = 0, 1, 2, 3, 4 and 5 are prepared using conventional melt-quenching technique.
2. The structure of the sample will be measured using X-ray diffraction technique to confirm the amorphous and crystalline phase of the sample.

3. The detailed structure of the bonding sample will perform using fourier transform infrared spectroscopy (FTIR) device.
4. The morphology of the sample will be shown via field emission scanning electron microscopy (FESEM).
5. The optical properties of the samples will be measured by using Ultra-Violet Visible (Uv-Vis) and photoluminescence (PL).

1.5 Important of the study

In this study, soda lime silicates (SLS) waste glass is a source of the silicate (SiO_2) in order to form a willemite sample. The making of the willemite based glass-ceramics from the silicate pure material was expensive to compare with the recycle soda lime silicate (SLS) glass. Beside the production of willemite from the waste glass will cut a cost, it also required low temperature for melting the glass component.
In Malaysia, 19,000 tons of wastes are produced every day, and a majority of that ends up in landfills. Malaysia currently has 230 landfill sites and 80% of them will reach maximum capacity within next two years, and with land for landfill site being at a premium, there is going to be a big problem to ours next generation.
In order to minimize the harmful effects on the environment caused by the landfill disposal solid waste, many of researchers have been studied and developed the methodologies for treatment and recycling the glass waste materials. One of the development methods was the addition or doping some oxide elements into the SLS glass structure (Sharma *et al*., 2007; Kadono *et al*., 2009) for the various applications.
In this research, ZnO had been chosen as an oxide to be added into the SLS glass to form a willemite. Besides that, erbium oxide has been chosen as a doped agent to improve mechanical and chemical properties of the sample that is suitable in luminescence for the optical application in this research.

1.6 Outline of thesis

The thesis arrangement is structured as follows. Chapter 1 gives an introduction of ZnO-SLS-Er_2O_3 glass and Er^{3+}-doped willemite glass-ceramics, the problem statements, the objectives, the scopes and the importance of the study. The theory of glass, glass-ceramics and previous works including past and current has been carried out by the other researchers are covered in Chapter 2. The apparatus, methodology and characterization of ZnO-SLS-Er_2O_3 glass and Er^{3+}-doped willemite glass-ceramics are explained in Chapter 3. The results concerning the effect of Er_2O_3 addition to the ZnO-SLS, progression of the sintering temperatures and optical properties of ZnO-SLS-Er_2O_3 glass and Er^{3+}-doped willemite glass-ceramics are analysed and discussed in Chapter 4. Finally, the conclusion and suggestion for the future works are presented in Chapter 5.

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