UNIVERSITI PUTRA MALAYSIA

STRUCTURAL, OPTICAL AND THERMAL PROPERTIES OF Sm_2O_3/Bi_2O_3-DOPED ZINC SILICATE GLASS CERAMICS FROM RICE HUSK ASH

AUWALU INUSA ABUBAKAR

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By

AUWALU INUSA ABUBAKAR

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

July 2017

DEDICATION

To my beloved parents Alhaji Inusa Abubakar and Malama Maryam Ahmad Gwadabe

For their absolute love and support

To my distinguish brothers, sisters and entire family
For making my life comfortable

To my dazzling wife Zainab Abdullahi Mukhtar
For her ultimate love and care

To all my cherished children
Yasin, Maryam, Almusari, Kanzillahi, Fatima and Suhaila
For their outstanding prayers

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

To all my noble lecturers
For helping me in way or other throughout this remarkable journey

Thank you all

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

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Chairman : Associate Professor Halimah Mohammed Kamari, PhD
Faculty : Science

The solid waste disposal has become a persistence problem in our communities. This evolves due to our rapid increase in population that leads to the growth of our industrial and agricultural sectors. A dramatic increase of solid waste deposition is experienced. Virtually, different collection of items is disposed, which change the compositional statues of our environments. Formally, solid waste disposal was carried out by incinerators technique. However, the burning of solid waste materials pollutes the surroundings. The community screams against the hazardous air pollution from incineration of waste materials. Hence, the technique changes to landfills solid waste disposal. Besides, this process tremendous number of problems is encompassing it, which changes the scientists and researchers way of thinking to the conversion of waste materials. Therefore, a subset of agricultural waste known as waste rice husk is converted to a useful material, known as white rice husk ash (WRHA) is used in carrying out this experimental work for this thesis. Conventional melt quenching technique was used to synthesis and characterize Sm^{3+}/Bi^{3+} doped zinc silicate derived from WRHA for the structural, optical and thermal properties. Some of the major findings were given as followed. At high sintering temperature, Porosities were decreased due to the diffusion of the ions into based precursor. The poly grains turned to aggregate with one another with increasing doping concentrations. The phonon was responsible for the conveying the heat energy. An increasing heat movement was noticed with growing samarium dopant concentrations. However, a fluctuated heat movement was observed due to the oxidation and de oxidation of Bi^{3+} ions. The decrement energy band gaps of the samples were occurring because of the conversion of bridging oxygen to non-bridging oxygen. Hence, in conclusion Samarium/Bismuth oxides doped zinc silicates were successfully synthesized via melt quenching technique. Porosity decreases with increase of Sm^{3+} / Bi^{3+} ions concentrations. The poly grains increase in size with increasing of Sm^{3+} / Bi^{3+} ions concentrations. The sample agglomerated more with 5 wt% increment of Sm^{3+} / Bi^{3+} ions concentrations. Thermal diffusivity increases with increase of samarium concentrations. Thermal diffusivity fluctuates with increase of bismuth concentrations. Optical band gap of

bismuth doped zinc silicate decreases with increase of bismuth concentrations. Optical band gap of samarium doped zinc silicate increases at 3 wt% and decreases with 5 wt% samarium concentrations. Refractive index decreases with Sm 3 wt% and increases with Sm 5 wt% while molar refraction and polarizability increase with samarium concentrations up to 3 wt% and decrease to 5wt%. Refractive index, molar refraction and polarizability decrease with increasing of bismuth concentrations. Finally, the outcomes of XRD, FESEM, and FTIR transmittance spectra showed the formation of zinc silicate phase in the precursor ZnO-WRHA glass ceramics. The FESEM micrograms indicated the formation of well-defined zinc silicate phase with highly agglomerated Samarium/Bismuth oxides -doped zinc silicate glass ceramics. The crystallite size acquired from XRD pattern differs in the range of 54-59 nm for samarium oxides -doped zinc silicate and 41-94 nm for bismuth oxides -doped zinc silicate. The presence of Zn-O and Si-O-Zn vibration bands in Infrared spectra shows the features of willemite phase formation. The UV-Visible analysis displays the value of optical band gap, which reduces with growing in doping concentrations, and the sintering temperature improves the willemite crystallinity in the precursor ZnO-WRHA glass ceramics. Furthermore, the luminescence spectra of glass-ceramics establish that the Sm^{3+}/ Bi^{3+} ions are diffusing into the glass-ceramic phase and through that improved the photoluminescence of the doped willemite glass-ceramics. Hence, the emission intensity of Zn_2SiO_4:Sm^{3+} phase showed red luminescence focused at 646.71 nm whereas the emission fixed at 621.57 nm developed from Zn_2SiO_4:Bi^{3+} phase matches to the $^4G_{5/2} – ^6H_{7/2}$ transition. The thermal diffusivities values grow with dopants proportions with the least value of 0.2039 - 0.1392 mm^2/s and highest value of 0.4375–0.2653 mm^2/s for Zn_2SiO_4:Sm^{3+}. Again, the thermal diffusivities values fluctuate with dopant concentrations with the least value of 0.2008–0.1383 mm^2/s and the highest value of 0.2329-0.1570 mm^2/s for Zn_2SiO_4:Bi^{3+}. For that reason, Zn_2SiO_4:Sm^{3+} glass-ceramics seems to be favorable for use in solid-state lasers as a promising phosphor material. On the other hand, Zn_2SiO_4:Bi^{3+} glass-ceramics suggests being favorable for use on radar screen as a promising phosphor material.

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

SIFAT STRUKTUR, OPTIK, DAN TERMA Sm_2O_3/ Bi_2O_3-DIDOP SERAMIK ZINK SILIKAT DARIPADA ABU SEKAM PADI

Oleh

AUWALU INUSA ABUBAKAR

Julai 2017

Pengerusi : Profesor Madya Halimah Mohammed Kamari, PhD
Fakulti : Sains

Masalah pelupusan sisa pepejal telah menjadi satu masalah yang berterusan dalam masyarakat kita. Perkembangan hal ini disebabkan oleh peningkatan populasi yang pesat yang membawa kepada pertumbuhan sektor perindustrian dan pertanian. Pemendapan sisa pepejal telah mengalami peningkatan mendadak. Secara kasarnya, pelbagai koleksi barangan yang berbeza telah dilupuskan, dimana ia mengubah komposisi pejal persekitaran kita. Secara rasmi, pelupusan sisa pepejal telah dijalankan menggunakan teknik insinerator. Walaubagaimanapun, pembakaran bahan sisa pepejal mencemarkan persekitaran. Komuniti masyarakat membangkang sekeras-kerasnya pencemaran udara berbahaya yang terhasil daripada pembakaran bahan-bahan buangan. Oleh itu, teknik ini berubah kepada tapak pelupusan sisa pepejal. Selain itu, proses ini merangkumi jumlah masalah yang besar, yang mengubah cara pemikiran para saintis dan penyelidik bagi penukaran bahan-bahan buangan. Oleh itu, satu subset sisa pertanian yang dikenali sebagai sisa sekam padi telah ditukarkan kepada bahan yang berguna, iaitu sisa abu sekam padi (WRHA) yang digunakan kerja-kerja eksperimen bagi tesis ini. Teknik pencairan pelindapkejutan secara konvensional telah digunakan bagi proses sintesis dan pencirian Sm^{3+}/ Bi^{3+} yang didop zink silikat berasal daripada WRHA bagi sifat-sifat struktur, optik dan terma. Antara penemuan utama adalah seperti berikut. Pada suhu pensinteran yang tinggi, keporosan berkurang disebabkan oleh penyebaran ion ke dalam dasar pelopor. Peningkatan konsentrasi bahan dop menukarkan butiran poli menjadi gumpalan antara satu sama lain. Fonon bertanggungjawab untuk menyalurkan tenaga haba. Peningkatan pergerakan haba telah disedari dengan peningkatan konsentrasi bahan dop samarium. Walaubagaimanapun, pergerakan haba yang berubah-ubah telah dilihat hasil pengoksidaan dan anti pengoksidaan Bi^{3+} ion. Penyusutan tenaga jurang jalur dalam sampel telah berlaku disebabkan oleh penukaran oksigen dengan penitian kepada oksigen bukan penitian. Oleh itu, sebagai kesimpulan Samarium/ Bismut oksida yang didop zink silikat telah berjaya disintesis melalui teknik pencairan pelindapkejutan. Keporosan berkurang dengan peningkatan konsentrasi ion Sm^{3+}/ Bi^{3+}. Saiz butiran poli meningkat dengan peningkatan konsentrasi ion Sm^{3+}/ Bi^{3+}. Penggumpalan sampel

bertambah bagi peningkatan 5 wt% konsentrasi ion Sm^{3+}/ Bi^{3+}. Kemeresapan haba meningkat dengan peningkatan konsentrasi samarium. Kemeresapan haba berubah-ubah dengan peningkatan konsentrasi bismut. Jurang jalur optik bagi bismut yang didop zink silikat berkurangan dengan peningkatan konsentrasi bismut. Jurang jalur optik bagi samarium yang didop zink silikat meningkat pada 3 wt% dan berkurang dengan 5 wt% konsentrasi samarium. Indeks biasan menurun dengan Sm 3 wt% dan bertambah dengan Sm 5 wt%, manakala molar pembiasan dan keterkutuban meningkat dengan kepekatan samarium sehingga 3 wt% dan berkurang untuk 5 wt%. Indeks biasan, molar pembiasan dan keterkutuban menurun dengan peningkatan konsentrasi bismut. Akhir sekali, hasil XRD, FESEM, dan pemindahan spektrum FTIR menunjukkan pembentukan fasa zink silikat di dalam pelopor seramik kaca ZnO-WRHA. Mikrogram FESEM menunjukkan pembentukan fasa zink silikat yang jelas dengan penggumpalan Samarium/ Bismut oksida -didop seramik kaca zink silikat yang tinggi. Saiz hablur yang diperolehi dari corak XRD berbeza dalam julat 54-59 nm bagi samarium oksida –didop zink silikat dan 41-94 nm bagi bismut oksida -didop zink silikat. Kehadiran jalur getaran Zn-O dan Si-O-Zn di dalam spektrum inframerah menunjukkan ciri-ciri pembentukan fasa wilemit. Analisis UV-Visible memaparkan nilai bagi jurang jalur optik, dimana ia berkurang dengan peningkatan konsentrasi bahan dop, dan suhu pensinteran meningkatkan penghabluran wilemit di dalam pelopor seramik kaca ZnO-WRHA. Tambahan pula, spektrum luminesens bagi seramik-kaca membuktikan bahawa Sm^{3+}/ Bi^{3+} ion meresap ke dalam fasa seramik-kaca dan meningkatkan fotoluminesens pendopkan wilemit seramik-kaca. Oleh itu, intensiti pelepasan bagi fasa Zn_2SiO_4: Sm^{3+} menunjukkan luminesens merah tertumpu di 646.71 nm manakala pelepasan tetap pada 621.57 nm yang dikembangkan dari fasa Zn_2SiO_4: Bi^{3+} berpadanan dengan transisi $^4G_{5/2}$ – $^6H_{7/2}$. Nilai peyerapan haba berkembang dengan perkadaran bahan dop dimana nilai paling sedikit adalah 0.2039-0.1392 mm^2/s dan nilai tertinggi adalah 0.4375-0.2653 mm^2/s untuk Zn_2SiO_4: Sm^{3+}. Sekali lagi, nilai penyerapan haba berubah-ubah dengan konsentrasi pendopan dengan nilai paling sedikit pada 0.2008-0.1383 mm^2/s dan nilai tertinggi pada 0.2329-0.1570 mm^2/s bagi Zn_2SiO_4: Bi^{3+}. Oleh kerana itu, Zn_2SiO_4: Sm^{3+} seramik-kaca sesuai untuk digunakan di dalam laser keadaan pepejal sebagai bahan fosfor yang mempunyai harapan yang cerah. Sebaliknya, Zn_2SiO_4: Bi^{3+} seramik-kaca adalah dicadangkan sesuai bagi penggunaan skrin radar sebagai bahan fosfor mempunyai harapan yang cerah.

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May Allah (S.W.A.) bless all the people that made this research work a success 'Amin'.

I certify that a Thesis Examination Committee has met on 14 July 2017 to conduct the final examination of Auwalu Inusa Abubakar on his thesis entitled "Structural, Optical and Thermal Properties of Sm_2O_3/Bi_2O_3-Doped Zinc Silicate Glass Ceramics Derived from Rice Husk Ash" 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 Doctor of Philosophy.

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

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Declaration by graduate student

I hereby confirm that:

- this thesis is my original work;
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This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) were adhered to.

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LIST OF ABBREVIATIONS

Zn_2SiO_4	Willemite
Zn_2SiO_4:Sm^{3+}	Samarium doped Willemite
Zn_2SiO_4: Bi^{3+}	Bismuth doped Willemite
Sm	Samarium
Bi	Bismuth
WRH	Waste rice husk
WRHA	White rice husk ash
SiO_2	Silicon dioxide
SiO_3	Silicon trioxide
ZnO	Zinc oxide
CaO	Calcium oxide
K_2O	Potassium oxide
Fe_2O_3	Ferric oxide
MnO	Manganese oxide
Uv	Ultraviolet
XRD	X-Ray diffraction
FTIR	Fourier transform infrared
FESEM	Field emission scanning electron microscopy
EDXRF	Energy dispersive X-ray fluorescence
TEM	Transmission electron microscopy
UV-Vis	Ultraviolet-Visible
E_{opt}	Optical band gap
PL	Photoluminescence
PVA	Polyvinyl alcohol
R_m	Molar refraction
V_m	Molar volume
α_m	Polarizability
ρ	Density
n_o	Refractive index

CHAPTER 1

INTRODUCTION

1.1 Research background

In recent years, solid waste disposal has become a persistence problem in our communities. This evolves due to our rapid increase in population that leads to the growth of our industrial and agricultural sectors. A dramatic increase of solid waste deposition is experienced. Virtually, different collection of items is disposed, which change the compositional statues of our environments. Formally, solid waste disposal was carried out by incinerators technique. However, the burning of solid waste materials pollutes the surroundings. The community screams against the hazardous air pollution from incineration of waste materials (Sembiring and Nitivattananon 2010). Hence, the technique changes to landfills solid waste disposal. Besides, this process tremendous number of problems is encompassing it, which changes the scientists and researchers way of thinking to the conversion of waste materials.

Figure 1.1: General solid wastes (Source: goo.gl/paFnx6)

Today in Malaysia, we can classify solids waste in to three categories domestic, industrial and agricultural wastes as shown in Figure 1.1. By using proper technique in treating these solid waste, some of them can be converted to be useful material example waste rice husk (WRH), as shown in Figure 1.3 which is a subset of agriculture solid wastes.

Figure 1.2: Agricultural solid wastes (https://goo.gl/TdRmPq)

The Figure 1.2 represents agricultural solid wastes. Waste rice husk can be converted to a useful material, known as white rice husk ash (WRHA) which is the main material used in carrying out this experimental work. White rice husk ash (WRHA) is one form of amorphous occurred at 700 ℃. WRHA simply consists of silicon dioxides (SiO_2) as major component with other slight constituents such as SiO_3, CaO, K_2O, Fe_2O_3, MnO, and CuO.

Figure 1.3: Waste rice husks (Source: goo.gl/P8zPmK)

WRHA can be used to make glass ceramic which is extremely inexpensive, reasonably hard, and chemically stable. Besides, WRHA glass ceramics also have excellent insulating properties and high mechanical properties. They are recognized as the radiation-sensitive dosimeter, especially glass ceramics doped with rare earth ions or transition metal ions. WRHA glass ceramics are attractive materials for the manufacturing of low-cost computer memory, catalytic converters, cell phones, and fluorescent (Noushad et al., 2012). In this thesis we focus on zinc oxides mixed with silica obtained from white rice husk ash to form zinc silicate willemite (glass ceramic) where later doped with samarium and bismuth oxides independently (Deepa et al., 2016). Zinc oxide is a semiconductor with an extensive energy band gap and having significant binding energy. In a glass ceramic network system, zinc oxide acts as a modifier with comprehensive applications. The purpose of this research is to explore the effect of samarium and bismuth oxides variations on the structural, optical and thermal diffusivity of zinc silicate (El Ghoul et al., 2012). An accurate replacement of zinc oxide by the samarium/bismuth oxide in the zinc silicate constituents will draw toward to form a better samarium/bismuth zinc silicate glass ceramics with a broad range of uses in optical materials for various technological and scientific applications. To the best of my knowledge, Sm_2O_3/Bi_2O_3 doped zinc silicate glass ceramic using white rice husk ash has not been done so far, which was additional motivation for this research work. Silicate and lead oxides are the illustrations of conventional glass ceramic formers while bismuth oxide is not, because of the small field strength of Bi^{3+} ions and high polarizability. Nevertheless, the bismuth oxide shows the most complicated function in glass ceramic formation. Bismuth oxide has an important quality of broad transmission range, nonpoisonous and high refractive index in the glass composition. The amorphous forms of silicate have extensive industrial applications. The bismuth-silicate glass ceramics have distinctive uses as optical amplifiers, third-order nonlinear susceptibility, low loss optical fibers, oscillators and infrared-transmitting materials. These glass ceramics also have high refractive index, high dielectric constant, high density, transmission in central of infrared region. The bismuth-silicate glass ceramics have vital benefits especially in sealing glass ceramics for metals, plasma panel displays, reflecting windows, thick film conductors, in the field of glass ceramics and electronic devices respectively. A considerable number of the studies were conducted on the structural and luminescence properties of doped zinc silicate such as europium doped zinc silicate manganese doped zinc orthosilicate and cerium doped zinc silicate glass ceramic. It is found that doped zinc silicate are excellent candidates for numerous industrial applications.

1.2 Problems statement

Within the series of conventional glass ceramics, WRHA glass ceramics have outstanding promising application due to their excellent glass forming character correlating to the discrete conventional glass ceramics (Hashmi et al., 2013). Besides, WRHA glass ceramic is accepted due to its fine mechanical and optical properties, such as high UV transparency, good chemical stability, low nonlinear refractive index and low thermal expansion coefficient leading to the strong thermal resistance (Sahu et al., 2015). WRHA glass ceramic also has large tensile fracture strength and good durability. The feasibility to regulate the physical properties such as density and refractive index of the glass ceramic is by the alteration of the glass ceramics composition (Eevon et al., 2016). A vigorous deliberation happened in the literature regarding the effect of doping of samarium/bismuth oxides into borate and telluride glass ceramic; however, the doping of samarium/bismuth oxides into WRHA glass ceramic is very few with no authoritative answers to the essential subject matter

(Matori et al., 2009). Despite, the diverse properties of WRHA glass ceramic such as good mechanical and high insulating properties have drawn attention of many researchers' due to the variety of technological and industrial applications (Patra et al., 2004). Lack of work for this glass ceramic had been investigated especially samarium and bismuth oxides doped zinc silicate (willemite) glass ceramic from white rice husk ash respectively. Therefore, an itemized study of the structural, optical and thermal properties of samarium/bismuth doped with WRHA glass ceramics are carried out and the results of this research are offered in this thesis.

1.3 Research objective

The research objectives are:

1. To synthesize of samarium and bismuth oxides, doped zinc silicate (willemite) derived from white rice husk ash.
2. To determine the optical absorption, optical band gap, refractive index, molar refraction, polarizability, excitation and emission spectra of samarium/bismuth oxides, doped zinc silicate ceramics derived from white rice husk ash.
3. To study the thermal properties of samarium/bismuth oxides zinc silicate ceramics derived from white rice husk ash.

1.4 Hypothesis

Zinc silicate (willemite) is a porous compound. The doping of samarium oxide and bismuth oxide will increase the crystal size structure, by the diffusion of electrons into the host matrix, which might reduce the porosity of the willemite. Further addition of the dopant may cause the structure to agglomerate. Concerning the optical properties samarium doped zinc silicate the optical band gap may increase up to a certain level then decrease to a certain level after the dopant addition. However, bismuth oxide doping zinc silicate may reveal the decrease in the optical band gap as the dopant concentration is increasing. The thermal properties may show the increase in thermal diffusivity with the increase in dopant concentration for samarium oxide doped zinc silicate. At the same time increasing in dopant concentration of bismuth oxide doped zinc silicate may fluctuates thermal diffusivities values.

1.5 Scope of the study

To achieve the objectives of the study, the scopes of the research are as follow:

1. The glass ceramic samples uses weight percentage pattern (70-X) $Zn_2$30SiO_4 XSm_2O_3 with X = 0, 1, 3 5 wt% and (60-X) $Zn_2$40SiO_4XBi_2O_3 with X = 0, 1, 3 and 5 wt% to prepare WRHA glass ceramics. By using conventional melt and quenching technique.
2. The structure of (70-X) $Zn_2$30SiO_4 XSm_2O_3 with X = 0, 1, 3 and 5 wt% and (60-X) $Zn_2$40SiO_4XBi_2O_3 with X = 0, 1, 3 and 5 wt% glass ceramic measures by X-ray diffraction technique to confirm the crystalline nature of the glass ceramic samples.
3. The energy dispersive X-ray fluorescence (EDXRF) measures the composition of the glass ceramic samples.
4. The Ultra-Violet Visible (UV-Vis) helps to measure the optical band gap. The Perkin Elmer LS 55 Fluorescence spectrometer is an instrument that allows measurement of photoluminescence (PL) means excitation and emission spectra of a sample with its electronic transitions.
5. The Archimedes' technique employed to measure the densities and combined with the obtained energy band gap to calculate the refractive index, molar refraction and polarizability of glass ceramic samples.
6. The Perkin Elmer TGA7 accomplishes TGA and DSC test under nitrogen gas at 1 atm. TGA test evaluate the mass changes of a sample with temperature. Moreover, NETZSCH-LFA 457 micro-flash device with an InSb sensor performed the thermal diffusivity test.

1.6 Importance of the study

Preparing of zinc willemite glass ceramic is quite expensive, and the most important component of willemite glass ceramic is silica. About 98.2% purity of the burnt white rice husk ash are silica as revealed by XRF results. Currently, Malaysia is positioned as twenty-five in the world rice producing countries (US dept. of agriculture, 2016). Most of the landfills in Malaysia are filled up with industrial and agricultural waste. In few years to come with rapid development in both agricultural and industrial sectors in Malaysia, this will increase the percentage of the waste production. To reduce landfill disposal of solid waste, conversion of waste rice husk into a useful material known as silica is a decent idea. Silica combined with the zinc oxide can form, zinc silicate (willemite) which have extensive industrial application (Sulowska et al., 2016). The research on the structural, optical and thermal properties of zinc silicate doped with rare earth and post-transitional elements are not well investigated. Therefore, additional research is significantly required to be performed in order to determine the important properties of zinc silicate doped with samarium/bismuth oxides.

1.7 Outline of thesis

The thesis structural arrangement is designed as follows, Chapter 1 narrates of zinc oxide and waste rice husk ash, zinc silicate (willemite) glass ceramics and Sm/Bi oxides doped zinc silicate, the objectives, the problem statements, the importance of the study and the scope of the research. The Chapter 2 corresponds with the recent and preceding works executed by other scholars of glass and glass-ceramic respectively. Chapter 3 explicitly deals with the theory of the electronics transition and the thermal movement through the entire samples. The methodological outline of study clearly states the characterizations of willemite glass-ceramic and willemite doped with samarium and bismuth oxides dopants all are in Chapter 4. Chapter 5 analyzes all the findings made in the research work such as XRD, FESEM, FTIR, EDX, TEM, UV-Vis, PL, T_g, DSC and thermal diffusivity for samarium and bismuth, oxides doped zinc silicate with different weight percentages sintered at 1000 ℃. Last but not the least Chapter 6 deals with the conclusion of the entire work and suggestions on how to carryout future research works.

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