UNIVERSITI PUTRA MALAYSIA

INFLUENCE OF Bi_2O_3 CONTENT ON THERMAL, STRUCTURAL AND OPTICAL PROPERTIES OF BISMUTH TELLURITE GLASS-CERAMICS BY CONTROLLED HEAT TREATMENT FOR OPTICAL APPLICATIONS

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By

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June 2021

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Faculty : Engineering

This dissertation has summarized the findings of the thermal, structural, and optical properties in xBi_2O_3-$5Na_2O$-$5TiO_2$-10ZnO-(80-x)TeO_2 based glass, where x= 5, 8, 10, 12, and 15mol% by using the melt quenching method. Multiples techniques have been carried on to characterize the influence of the Bi_2O_3 content on the thermal, structural, and optical properties of the bismuth tellurite based glass. The thermal properties have been analyzed by differential scanning calorimetry to identify the glass transition temperature, T_g, onset crystallization temperature, T_x, and crystallization temperature, T_c. The increasing Bi_2O_3 content causes the T_g to decrease due to the decomposition of strong TeO_4 units into TeO_{3+1} polyhedra and TeO_3 units and the replacement of strong Te-O-Te bonds by the formation of weaker Te-O-Bi bonds and Bi-O-Bi bonds. The formation of BiO_3 and BiO_6 can be observed when reaching an optimum percentage of Bi_2O_3. The Bi-rich phase will enhance the rate of nucleation and crystals growth rate which improve the crystallization tendency of the glass samples. Thus, a greater tendency for the glass ceramics transformation. The high content of Bi_2O_3 has caused the glass ceramics to lose transparency due to the oversize crystalline growth. On the other hand, the structural changes based on the variation of composition also investigated by X-Ray diffraction (XRD), Raman spectroscopy, and Fourier transform infrared (FTIR). The results from the structural analysis have proven the formation of BiO_3 and BiO_6 units. The formation of BiO_3 and BiO_6 units with more open structure has increased with the increasing percentage of Bi_2O_3. The more open structures have further improved the rate of nucleation and crystal growth within the glass matrix. Thus, the higher content of Bi_2O_3 has favoured the glass ceramics transformation. As a result of the heat treated sample glasses, the Bi_2O_3 content was strongly affecting the transparency of the resultant glass-ceramics due to the crystallization behaviour of the Bi_2O_3. The types of crystal formed within the glass matrix after controlled heat treatment were identified through X-ray diffraction (XRD). The $Bi_2O_{3.96}$ crystal started to form within the glass matrix with a lower Bi_2O_3 content and the transparency of the glass-ceramics was retained in these samples. Sample glass-ceramics were started to turn opaque on the samples with high Bi_2O_3 content with the formation of β-Bi_2O_3 crystals. Besides that, the vibration modes

of the structural units and the functional group of the elements before and after the controlled heat treatment were identified by Raman and FTIR. In addition, the optical properties including the direct and indirect energy bandgap, E_g which induced optically by the incident photons of the amorphous nature before controlled heat treatment, and the crystalline structure after the heat treatment were investigated through the UV-Visible absorption spectrophotometer. The energy bandgap, E_g has decreased due to the high concentration of NBO within the glass network which increases the excitation tendency of the electrons to the conduction band as the content of Bi_2O_3 increased. The sharp absorption edge can be observed in the heat treated glass and the sample glass ceramics with 12mol% and 15mol% Bi_2O_3 content have the most significant absorption edge as compared with the lower Bi_2O_3 content. The wavelength transmission in visible wavelength were extremely low in sample glass ceramics with 12mol% and 15 mol% of Bi_2O_3 content. Thus, the transparency of these sample glass ceramics were extremely low. The optical bandgap also reduced more in sample glass ceramics with greater Bi_2O_3 content as compared with the sample glasses. Most importantly, the transparency of the bismuth tellurite glass-ceramics has to be maintained above 50% for optical applications. The transparency of the samples started to decreased and turned opaque by the increasing Bi_2O_3 content and longer heat treatment period. The crystal growth of the tellurite glass-ceramics also has been investigated by using the Field Emission Scanning Electron Microscope. In conclusion, the optimum Bi_2O_3 content was 10mol% which has a significant crystallization tendency that showing 2 significant crystallization peaks. The 10 mol% glass ceramics having optimized glass ceramics properties with an excellent transparency at the same time. However, the heat treatment period has to be monitored to prevent the overgrowth of crystal which directly affecting the transparency of glass ceramics.

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

PENGARUH KANDUNGAN Bi_2O_3 TERHADAP TERMAL, HARTA STRUKTUR DAN OPTIK TERHADAP SERAMIK KACA BSMUT TELLURIT DENGAN RAWATAN PANAS YANG DIKAWAL UNTUK KEGUNAAN OPTIK

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Disertasi ini telah meringkaskan penemuan sifat termal, struktur, dan optik dalam kaca xBi_2O_3-$5Na_2O$-$5TiO_2$-10ZnO-(80-x)TeO_2, di mana x = 5, 8, 10, 12, dan 15 dengan menggunakan kaedah peleburan lebur. Beberapa teknik telah dijalankan untuk mencirikan pengaruh kandungan Bi_2O_3 pada sifat termal, struktur, dan optik kaca berasaskan bismut telurit. Sifat termal telah dianalisis dengan permeteran kalorimetri pengimbasan pembezaan untuk mengenal pasti suhu peralihan kaca, T_g, permulaan suhu penghabluran, T_x, dan suhu penghabluran, T_c. Kandungan Bi_2O_3 yang semakin meningkat menyebabkan T_g menurun kerana penguraian unit TeO_4 yang kuat menjadi unit polyhedral TeO_{3+1} dan TeO_3 dan penggantian ikatan Te-O-Te yang kuat dengan pembentukan ikatan Te-O-Bi dan Bi-O-Bi yang lebih lemah. Pembentukan BiO_3 dan BiO_6 dapat diperhatikan ketika mencapai peratusan Bi_2O_3 yang optimum. Fasa Bi kaya akan meningkatkan kadar nukleasi dan kadar pertumbuhan kristal yang meningkatkan kelegapan penghabluran sampel kaca. Oleh itu, kelegapan yang lebih besar untuk transformasi seramik kaca. Kandungan Bi_2O_3 yang tinggi telah menyebabkan seramik kaca kehilangan kelutsinaran kerana pertumbuhan kristal yang terlalu besar. Sebaliknya, perubahan struktur berdasarkan variasi komposisi juga diselidiki oleh difraksi sinar-X (XRD), spektroskopi Raman, dan inframerah transformasi Fourier (FTIR). Hasil dari analisis struktur telah membuktikan pembentukan unit BiO_3 dan BiO_6. Pembentukan unit BiO_3 dan BiO_6 dengan struktur yang lebih terbuka telah meningkat dengan peningkatan peratusan Bi_2O_3. Struktur yang lebih terbuka telah meningkatkan kadar nukleasi dan pertumbuhan kristal dalam matriks kaca. Oleh itu, kandungan Bi_2O_3 yang lebih tinggi telah meningkatkan prestasi transformasi seramik kaca. Akibat dari gelas sampel yang dirawat dengan panas, kandungan Bi_2O_3 sangat mempengaruhi kelutsinaran seramik kaca yang terhasil kerana tingkah laku penghabluran Bi_2O_3. Jenis kristal yang terbentuk di dalam matriks kaca setelah rawatan haba terkawal dikenal pasti melalui pembelauan sinar-X (XRD). Kristal $Bi_2O_{3.96}$ mula terbentuk dalam matriks kaca dengan kandungan Bi_2O_3 yang lebih rendah dan kelutsinaran seramik kaca dipertahankan dalam sampel ini. Sampel kaca seramik mula menjadi legap pada sampel dengan kandungan Bi_2O_3 yang

tinggi dengan pembentukan kristal β-Bi_2O_3. Selain itu, mod getaran unit struktur dan kumpulan berfungsi elemen sebelum dan selepas rawatan haba terkawal dikenal pasti oleh Raman dan FTIR. Di samping itu, sifat optikal termasuk jalur jurang terus dan tidak langsung, E_g yang disebabkan secara optik oleh foton amorf sebelum rawatan haba terkawal, dan struktur kristal selepas rawatan haba disiasat melalui spektrofotometer penyerapan UV-Visible. Jalur jurang, E_g telah menurun kerana kepekatan NBO yang tinggi di dalam rangkaian kaca yang meningkatkan kelegapan pengujaan elektron ke jalur konduksi ketika kandungan Bi_2O_3 meningkat. Pinggir penyerapan yang tajam dapat dilihat pada kaca yang dirawat panas dan sampel seramik kaca dengan kandungan Bi_2O_3 dalam 12mol% dan 15mol% mempunyai kelebihan penyerapan yang paling ketara berbanding dengan kandungan Bi_2O_3 yang lebih rendah. Penghantaran panjang gelombang dalam panjang gelombang nampak sangat rendah pada seramik kaca sampel dengan kandungan 12mol% dan 15 mol% Bi_2O_3. Oleh itu, kelutsinaran seramik kaca sampel ini sangat rendah. Jurang jalur optik juga menurun lebih banyak dalam seramik kaca sampel dengan kandungan Bi_2O_3 yang lebih besar berbanding dengan gelas sampel. Yang paling penting, kelutsinaran seramik kaca bismuth tellurit harus dijaga untuk aplikasi optik. Kelutsinaran sampel mula menurun dan menjadi legap dengan peningkatan kandungan Bi_2O_3 dan tempoh rawatan haba yang lebih lama. Pertumbuhan kristal seramik kaca bismut tellurit juga telah diselidiki dengan menggunakan Mikroskop Elektron Pengimbasan Pelepasan Lapangan. Kesimpulannya, kandungan Bi_2O_3 optimum adalah 10mol% yang mempunyai kecenderungan penghabluran bererti yang menunjukkan 2 puncak penghabluran yang bererti. Seramik kaca yang berkandungan 10 mol% bismut mempunyai sifat seramik kaca yang dioptimumkan dengan kelutsinaran yang sangat baik pada masa yang sama. Walau bagaimanapun, tempoh rawatan haba harus dipantau untuk mengelakkan pertumbuhan kristal yang berlebihan yang secara langsung mempengaruhi kelegapan seramik kaca.

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LIST OF ABBREVIATIONS

DSC	Differential Scanning Calorimetry
XRD	X-Ray Diffraction
FTIR	Fourier Transform Infrared
FESEM	Field Emission Scanning Electron Microscope
UV-Vis	Ultraviolet-Visible
T_g	Glass transition temperature
T_x	Onset crystallization temperature
T_c	Crystallization temperature
T_m	Melting temperature
IR	Infrared
NLO	Nonlinear optics
SHG	Second harmonic generation
HMO	Heavy metal oxides
UV	Ultraviolet
NIR	Near infrared
MIR	Mid infrared
T	Temperature
mol%	Molarity percentage
ZnO	Zinc oxides
TeO_2	Tellurium oxides
Na_2O	Sodium oxides
TiO_2	Titanium oxides
Bi_2O_3	Bismuth oxides
Bi^{3+}	Bismuth ions
BiO_3	Oxobismuthinolate oxide

E_{opt}	Optical band gap
$NaCO_3$	Sodium carbonate
TGA	Thermo-Gravimetric Analysis
λ	Wavelength
V	Volts
A	Ampere
Al_2O_3	Aluminium oxides
ρ	Density
Vm	Molar volume
NBO	Non-bridging oxygen
BO	Bridging oxygen
S	Glass stability
TBP	Trigonal bipyramid
TP	Trigonal pyramid
Ti^{4+}	Titanium ions
χ^3	Nonlinear susceptibility
Er^{3+}	Erbium ions
Yb^{3+}	Ytterbium ions
Tb^{3+}	Terbium ions

CHAPTER 1

INTRODUCTION

1.1 Aims and motivation

In recent years, the direction of development in the communication system has shifted slowly from the ordinary electrical communication system using copper wires as a transmission medium for signal transmission to optical communication system. The reasons for this trend are due to the limitation on the capacity and speed of the transmission when copper wires are used as a transmission medium in the communication system. However, this problem can be solved by the invention of the optical communication system that uses optical fibre as the transmission medium of the communication system. The benefits of using optical fibre as a transmission medium are to increase the capacity and the speed of signal transmission within the communication system.

Essentially, a comprehensive optical communication system is a built using multiple optical components, such as optical switches, optical amplifiers, and optical fibre. For the design of fibre-based and optical waveguides material, the characteristics of the glass material in thermal stability and crystallisation properties are important as crystal growth within the glass materials due to reheating during laser operation will lead to a linear loss through light scattering centre in the waveguide, which potentially negates the signal gain [1]. In order to optimise data transmission, it is vital that the manufacturing materials used for optical components have a good refractive index, transmittance, and non-linear optical properties. Due to this reason, many efforts have been made to improve the data or signal transmission on various types of glasses from the perspective of capacity, and low loss and wide transmission window from ultraviolet (UV) to near-infrared (NIR) [2]. Tellurite-based glasses have stirred wide-ranging interest in the field of photonic for their high refractive index, low phonon energy, good thermal stability, and excellent chemical durability. The low phonon energy of the tellurite glass minimises the non-radiative losses of the material through the reduction of the non-radiative transition probability of the ions in the glass [3][4]. Most importantly, linear and non-linear optical (NLO) properties of the tellurite glasses are excellent in the third-order optical non-linearity and ultrafast non-linear optical response [5]. Moreover, the nonlinear refractive index of tellurite-based glasses is higher than other types of glasses, such as silicate, borate, or germanate glasses [6]. This has made tellurite-based glass increasingly prominent as materials for laser, non-linear optics, and optical communication [7].

The significant non-linear optical properties of TeO_2-based glasses are mainly due to the high polarisability of the lone pair electron in the 5s orbital of the tellurium atom [8]. Tellurite glass is a type of conditional glass former in which tellurium is unable to form glass by a single oxide. The reason for this is because pure TeO_2 is unstable and will quickly crystallise due to the lone pair electrons, which are present at the equatorial position of the TeO_4 units. Due to this reason, the structural rearrangement of these units during the glass formation process is strictly limited. Thus, the modifier oxides, such as alkali, alkaline, or heavy metal oxides, have to be added to the composition in order to improve the glass-forming ability of the tellurium oxides. The modifier oxides that are generally recommended to avoid crystallisation by enhancing the glass stability are ZnO and Na_2O [6][9]. Additionally, the great non-linear optical properties due to the high polarisability of Te^{4+} can be further enhanced by simply adding the modifier oxides that involve the highly polarisable cations in the heavy p elements. The modifier oxides of high atomic weight, such as lead oxides (PbO) and bismuth oxides (Bi_2O_3), are also able to enhance the refractive index and the transmission windows of the tellurite-based glasses, which extend the infrared (IR) spectrum. In the current development of glasses, glasses have been mainly divided into two types: amorphous glasses and glass-ceramics. Amorphous glass's characteristic is that the arrangement of the glass structure and the network fully random. As for glass-ceramics, the organized crystal structures and networks can be observed within the amorphous glass network. There are few great potentials on the tellurite glass-ceramics, which have greater mechanical properties, refractive index, and nonlinear properties as compared to the amorphous tellurite based glass. Thus, the potential of tellurite glass-ceramics on the application of optical devices is significant [10][11].

High refractive index tellurite glass-ceramics can be obtained when the refractive index of the crystalline phases formed within the glass network structures is similar to the refractive index of the glass phases within the glass matrix. This phenomenon can be explained by the reduction of scattering within the glass matrix. When the refractive index of the phases within the glass matrix is similar, the scattering loss when the incident light travels from one phase to another phase can be reduced. Thus, the glass refractive index can be modified by converting the amorphous glass into glass-ceramics. On the other hand, the formation of the crystal phases has broken the long-range isotropic glass network of the tellurite based glass. The nonlinear optical properties such as second harmonic generation (SHG) will be enhanced due to the formation of the crystal in the tellurite glass-ceramics [10]. A nonlinear crystals can be used for the conversion of energy in an electromagnetic wave with a certain value of frequency or multiples electromagnetic waves at different frequencies to energy in an electromagnetic wave at a different frequency.

By referring to the earlier work that has been done by previous researchers, Bi_2O_3 is a great modifier oxide that is able to improve the crystallisation behaviour of the tellurite-based glass [10]. Thus, Bi_2O_3 has been chosen as the varying modifier oxide in this project to observe the difference in structural, optical properties, and crystallisation behaviour between the tellurite-based glass and the tellurite glass-ceramics by converting the amorphous tellurite-based glass into tellurite glass-ceramics. The conversion of the amorphous tellurite glass into tellurite glass-ceramics is done by control heat treatment. Controlled heat treatment is a doublestep heat treatment, which is the nucleation and crystals growth. There is a difference in the microstructure that can be observed by controlled heat treatment as compared to the single-step heat treatment [11]. The purpose of using controlled heat treatment is to maintain the crystal size below 1 µm, which controls the transparency of the tellurite glass-ceramics.

1.2 Problem statement

As mentioned from the part of aims and motivation, an optical communication system should have characteristics, such as high speed and low loss, to transmit a huge amount of data at one time, especially with long-distance communication. Thus, the glass materials used should have characteristics, such as low absorption of light and wide transmission bandwidth.

Other than the novel glass of silicate glass composition, there are a lot of other more optical flexible glass compositions that can be obtained with a high refractive index, transparent window, and great linear and nonlinear optical properties, such as heavy metal oxides, fluorides, and chalcogenides [12]. In this project, tellurium oxides, which is able to fulfill the characteristics that have been mentioned, will be used as the glass former in our glass composition.

First, significant transparency is an important characteristic of the medium for optical applications. Thus, the transparency of the glass samples needs to be maintained even the sample glasses have undergone the crystallisation process through heat treatment, which converts from glass to glass-ceramics. In other words, the crystal size needs to be controlled for the prevention of the overgrew of the crystals within the glass-ceramics, which causes the scattering of light that lead to the opacity of the sample glasses [10].

Next, the refractive index of the glass material needs to be significant because the refractive index is the fundamental criterion of an optical material. The high refractive index enables the material to have high polarisability and high potential to act as a low loss optical waveguide through total internal reflection with an appropriate combination of material [13][14]. Material with great polarisability is expected to have significant nonlinear optical properties, which, in turn, have a large nonlinear susceptibility, χ [13]. The nonlinear coefficient of an optical material plays an important role in various optical applications, such as optical lenses, optoelectronics, and optical communication. Since tellurite glass is considered as a high refractive index glass, the addition of the high refractive index heavy metal oxides (HMO), Bi_2O_3 as a glass modifier will further enhance the polarisability of the high polarisable tellurite glass [15]. However, the addition of Bi_2O_3 also able to enhance the crystallisation tendency of the tellurite-based glass at the same time [10]. Thus, the thermal properties, network structure, and tendency of crystallization by the influence of Bi_2O_3 addition have to be investigated.

Lastly, the optical induced transition can be observed through the absorption edge from the UV-Visible absorption investigation [16]. The band structure and the energy gap of the non-crystalline and crystalline material can be determined through the absorption investigation. Thus, the changes in the bandgap and energy structure of the bismuth tellurite glass representing non-crystalline material before controlled heat treatment and bismuth tellurite glass-ceramics, which crystallised after the heat treatment can be investigated.

1.3 Objectives

In this thesis, the thermal, structural, and optical properties of the bismuth tellurite glass with different percentages of bismuth oxides, Bi_2O_3, before and after controlled heat treatment will be investigated. The objectives of this project can be concluded as follow:

1. To evaluate the optical transparency of the bismuth tellurite glass-ceramics even after the controlled heat treatment.
2. To analyse the thermal properties, structural network, and the tendency of crystallisation of the bismuth tellurite glass with different percentages of the bismuth oxides, Bi_2O_3 content.
3. To investigate the optical induced bandgap and energy structure of the bismuth tellurite glass before and after heat treatment.

1.4 Scope

In this thesis, the fabrication of the glass, heat treatment, and various types of testing of the sample glasses will be carried out to perform the best results of this project. The scope of this project can be summarised as follow:

1. The bismuth tellurite glasses with different percentages of Bi_2O_3 content will be fabricated by using the melt quenching method.
2. The thermal properties of the sample glasses with different percentage of Bi_2O_3 content will be performed by differential scanning calorimetry (DSC).
3. Heat treatment will be performed based on the DSC results, and the temperature for the heat treatment will be referring to the glass transition temperature (T_g), onset crystallisation temperature (T_x), and crystallisation temperature (T_c).
4. The degree of crystallinity of the sample glasses before and after heat treatment will be performed by x-ray diffraction.
5. The structural properties of the sample glasses before and after heat treatment will be performed by Raman spectroscopy and Fourier-transfer infrared spectroscopy (FTIR)
6. The optical properties of the sample glasses before and after heat treatment will be performed by UV-Vis spectroscopy.

1.5 Report structure

An overview of the structure of this report is presented in this chapter. The problem statement in this chapter has listed the problems faced in the current technologies. The objective was the purpose of carrying out this project and also as guidance or directory of the project. Meanwhile, the scope was the solutions that how the problem stated able to be solved. Furthermore, the report structure will be summarized the content or idea that will be discussed in each of the chapters.

Chapter 2 has reviews the research that have been done by the other researchers. The idea and the techniques that have been carried out by the researchers have been extracted and compounded as references for the project. The idea and techniques have been extracted here, including glass properties, characteristics of glass-ceramics, structural, thermal, and optical properties of the compositions. Other than that, the comparison among the results by the researchers also has been done to extract the best solutions or techniques to proceed with the project.

Chapter 3 describes the basic steps or procedures that has been carried out in this research. First, this chapter has described the preparation of the sample glasses, which includes chemical mixing and glass melting process. Next, the conversion of sample glasses into glass-ceramics by using controlled heat treatment also has been discussed in this chapter. Lastly, the characterisation of thermal, structural, and optical properties of the sample glass before and after heat treatment also has been described in this chapter.

Chapter 4 discusses the results that have been obtained in this project. First of all, this chapter has shown the transparency of the sample glasses before and after heat treatment. Subsequently, the degree of crystallinity of the heat-treated glass samples also has been described in this chapter. At the end of this chapter, the results of the characterization of thermal, structural, and optical properties also have been discussed in this chapter.

Chapter 5 has concludes and summarises the research on the changes in the sample glasses before and after heat treatment in the perspective of transparency, degree of crystallinity, thermal, structural, and optical properties.

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