UNIVERSITI PUTRA MALAYSIA

DIELECTRIC AND OPTICAL PROPERTIES OF B2O3 – TeO2 – Sm2O3 GLASS SYSTEM

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DIELECTRIC AND OPTICAL PROPERTIES OF B_2O_3 – TeO_2 – Sm_2O_3 GLASS SYSTEM

By

CHE ZURAINI BINTI CHE AB RAHMAN

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

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DEDICATIONS

For my beloved parents,

CHE AB RAHMAN BIN CHE MAHMOOD
and
LATIFAH BT JAAFAR.

And also to my siblings and family.

To all my helpful lecturers.

To all of my dearest friends.

With all of my hearts, I thank you for all of your supports.

May Allah bless all and ease everything.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

DIELECTRIC AND OPTICAL PROPERTIES OF $B_2O_3 – TeO_2 – Sm_2O_3$ GLASS SYSTEM

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November 2014

Chairman : **Associate Professor W. Mohamad Daud W. Yusoff, PhD.**

Faculty : **Science**

Glasses containing ternary system $(B_2O_3)_{30}$ $(TeO_2)_{70-x}$ $(Sm_2O_3)_x$ and $(B_2O_3)_{30-x}$ $(TeO_2)_{70}$ $(Sm_2O_3)_x$ were prepared by melt-quenching technique over a wide range of composition (x = 0.3, 0.5, 0.7, 1.0 and 1.2 mol %) denoted as BTS1, BTS2, BTS3, BTS4, BTS5 and BTS6, BTS7, BTS8, BTS9, BTS10 respectively. The present study characterize Sm_2O_3 doped TeO_2-B_2O_3 glasses composition, physical, structural, optical and dielectric properties as a function of frequency and temperature as well as the effect of the rare earth ions (Sm^{3+} ions) introduced into these glasses systems. The present investigation on the dielectric properties is useful to understand the basic mechanism of the polarization process, and the parameters necessary for the basic understanding of the physical grounds of optical properties. The structural changes were studied by X-Ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy and Differential Thermal Analysis (DTA) curve. All glasses studied in the present work exhibit amorphous materials. Network units existed in Sm^{3+} doped glasses and Sm^{3+} ions existed as network modifiers. The higher concentration of Sm^{3+}, the more units of TeO_3 groups transform to TeO_4 groups in BTS1 - BTS5, more units deformed TeO_4 group were transform to TeO_3 groups in BTS6 - BTS10 and the transformation of some BO_4 unit into BO_3 units. The optimum Sm^{3+} concentration was about 1.0 mol% (BTS4 and BTS9) for this glasses system due to the Sm^{3+} concentration quenching. The density (ρ) decreases and molar volume (V_m) were reversed to ρ, attributed to non-bridging oxygen as the addition of samarium oxide (Sm) content. The addition of samarium oxide ($4f^5$) indicates strong bonding between the components and enhances the glass formation ability (GFA). The optical and dielectric properties were studied in the frequency ranging from 10^{-2} Hz to 10^6 Hz, at temperature ranging from 373 K to 493 K. The dielectric properties (dielectric constant, ε', and dielectric loss factor, ε'') show a larger value at lower frequencies (below 100 Hz) and higher temperatures (above 413 K). The graphs were fitted using Cole-Cole functions and Quasi dc models. Activation energy obtained from the master curve graph was found to decrease as Sm_2O_3 content increased. The optical properties show that the indirect band gap values are decreasing with Sm_2O_3 content

up to BTS4 (2.24 to 2.12 eV) and BTS9 (2.32 to 2.18 eV) attributed to increase in degree of disorder in the system, direct consequence of the increases of non-bridging oxygen in the system.

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

PENCIRIAN DIELEKTRIK DAN OPTIKAL BAGI SISTEM KACA B_2O_3 – TeO_2 – Sm_2O_3

Oleh

CHE ZURAINI BINTI CHE AB RAHMAN

November 2014

Pengerusi : **Professor Madya W. Mohamad Daud W. Yusoff, PhD.**

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Kaca mengandungi sistem pertigaan $(B_2O_3)_{30}$ $(TeO_2)_{70-x}$ $(Sm_2O_3)_x$ dan $(B_2O_3)_{30-x}$ $(TeO_2)_{70}$ $(Sm_2O_3)_x$ telah disediakan melalui teknik sepuh lindapan merangkumi komposisi (x = 0.3, 0.5, 0.7, 1.0 dan 1.2 mol %), masing-masing dilabel sebagai BTS1, BTS2, BTS3, BTS4, BTS5 dan BTS6, BTS7, BTS8, BTS9, BTS10. Kajian ini mencirikan Sm_2O_3 ditambah pada B_2O_3-TeO_2 komposisi gelas, fizikal, struktur, sifat-sifat optik dan dielektrik sebagai fungsi frekuensi dan suhu serta kesan ion nadir bumi (ion Sm^{3+}) diperkenalkan ke dalam sistem kaca ini. Penyelidikan pada sifat-sifat dielektrik adalah berguna untuk memahami mekanisme asas proses polarisasi, dan parameter-parameter yang perlu bagi pemahaman asas mengenai sifat-sifat fizikal optik. Perubahan pada struktur kaca dikaji melalui Pembelaun sinar-X (XRD), spektroskopi infra merah (FTIR) dan analisis perbezaan lengkungan terma (DTA). Semua kaca yang dikaji di dalam kajian ini bersifat bahan amorfus. Unit rangkaian wujud di dalam kaca campuran Sm^{3+} dan Sm^{3+} wujud sebagai rangkaian pengubahsuai. Semakin tinggi kepekatan Sm^{3+}, semakin banyak unit TeO_3 ditukarkan kepada unit TeO_4 bagi gelas BTS1 - BTS5, semakin banyak unit TeO_4 cacat ditukarkan kepada unit TeO_3 bagi gelas BTS6 - BTS10 dan pertukaran unit BO_4 kepada unit BO_3. Kepekatan optimum bagi Sm^{3+} di dalam sistem kaca ini adalah sebanyak 1.0 mol % (BTS4 dan BTS9). Ketumpatan (ρ) berkurangan dan isipadu molar (V_m) berlawanan dengan ρ diukur berkait melalui oksigen tak bersambungan apabila samarium oksida ditambah. Penambahan samarium oksida ($4f^5$) menunjukkan ikatan yang kuat antara komponennya dan menggalakkan keupayaan pembentukan kaca (GFA). Pemalar-pemalar optikal dan dielektrik dikaji di dalam julat frekuensi 10^{-2} Hz hingga 10^6 Hz, pada julat suhu 373 K hingga 493 K. Pemalar-pemalar dielektrik (ε' dan ε'') menunjukkan nilai yang besar pada frekuensi yang rendah (100 Hz kebawah) dan pada suhu yang tinggi (413 K keatas). Graf diplot menggunakan model Cole-Cole dan model Quasi-dc. Tenaga pengaktifan yang dicerap daripada graf lengkungan utama didapati berkurangan apabila komposisi Sm_2O_3 bertambah. Pemalar-pemalar optikal menunjukkan sela jalur tidak terus semakin berkurang dengan penambahan komposisi Sm_2O_3 sehingga BTS4 (2.24 hingga 2.12 eV) dan BTS9 (2.32 hingga 2.18 eV) dianggap berikutan dengan

peningkatan pada darjah tak tertib dalam sistem, sejajar dengan peningkatan oksigen tak bersambungan di dalam sistem kaca ini.

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I certify that a Thesis Examination Committee has met on 14 November 2014 to conduct the final examination of Che Zuraini binti Che Ab Rahman on her thesis entitled "Dielectric and Optical Properties of $B_2O_3 - TeO_2 - Sm_2O_3$ Glass System" in accordance with 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.

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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 Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABREVIATIONS AND SYMBOLS

Abbreviations	Description
XRD	X-ray diffraction
FTIR	Fourier transform infrared spectroscopy
DTA	Differential thermal analysis
GFA	Glass forming ability
RED	Rare earth doped
RE	Rare earth
Sm	Samarium
IR	Infrared

Symbols	Description
ε’	Dielectric constant
ε’’	Dielectric loss factor
ΔT	Difference between Tc and Tg
ρ	Density
V_m	Molar volume
E_{opt}	Energy band gap
E_a	Activation energy
T_g	Glass transition temperature
T_c	Crystallization temperature
T_m	Melting temperature

CHAPTER 1

INTRODUCTION

1.1 General Introduction

The first glass objects dated back to before 2000 BC. Throughout history, glass has been used to make ornamental and decorative objects. In addition, it has been used for useful objects such as windows, containers, optical lenses, and glass fibers.

Glass is an amorphous solid, and fused mixtures of inorganic oxides which are cooled to a solid state without crystallization. Its molecules have a disordered arrangement, but there is enough cohesion to produce rigidity. The majority of glass seen in everyday life is transparent, but can also be translucent or opaque. The main ingredient in glass is silica. Silica can be melted to form fused silica. However, silica has too high melting point to fuse. Therefore, a flux is added which lowers the melting point. Soda is an example of a flux. Once we melt silica and let it cool, we will get glass. However, it will not be strong or stable. To make glass stable, a stabilizer is added. Examples of stabilizers are limestone, magnesia, and zirconia. Silica can be combined with other material to form other generic forms of glass that are more widely used. The range of melting points for glass, depending on its composition, is 500 to 1650 °C. Glass is a poor conductor of heat and electricity and is therefore used as an insulator.

A study of dielectric materials has been experimented at early 20th century ago. The dielectric material is a type of materials which have the specific characteristic, such as behave like a poor conductor material in many situations. The prime asset of high-dielectric-constant substances is the fact that they make possible the manufacturing of high-value capacitors with small physical volume. While for engineering members, they are interested to study about the dielectric properties of materials because a dielectric material is a substance that is a poor conductor of electricity, but an efficient supporter of electrostatic fields. This material ensures the poor conductor material to have a high resistance, high dielectric ability and a low dielectric loss tangent (Pollock *et al*., 1980).

Throughout recent years, there has been considerable interesting semiconducting glasses in which tellurium oxide is a glass forming factor and known as a conditional glass former, in the sense that it needs a modifier (such as alkaline, alkaline earth and transitional metal oxides or other glass modifiers) in order to form the glassy state easily (Khaled *et al*., 1994). Distinguishing factor of telluride glass matrix is that the tellurium atoms have unshared pairs of electrons which do not take part in bonding and they have a weaker Te-O bond. These bonds are easily broken and this is an advantage of accommodating rare earth ions into this glass (Sabry and Samanoudy, 1995; Tuller *et al*., 1980).

Telluride glasses possess an excellent physical properties for instance high refractive index, low melting temperature, high dielectric constant and high infrared transmission (Tanaka *et al*., 1988). Due to that, study on telluride based glasses have been carried out in recent years on a variety of inorganic glasses by a number of researchers yielding valuable structural information due to their interesting optical, electrical and magnetic properties. They have promising applications in the nonlinear optical devices such as optical switching, optical memory, gas sensors and also found that telluride glasses are usually good insulators (El-Mallawany, 2000; Hampton *et al*., 1987).

Tellurite glasses are considered as one of the best hosts for doping with rare earth elements. It is a good candidate for practical laser applications because of low crystallization rate, excellent transparency in a wide spectral range (3–18μm), good mechanical stability and chemical durability (Weber *et al*., 1981; Nii *et al*., 1998; Wang *et al*., 1994). The optical properties of tellurite glasses doped with rare earth have been investigated by several groups (Wang *et al*., 1994; Xu *et al*., 2005; Romanwski *et al*., 1988). TeO_2-based glasses were most studied in the last 10 years, considering the scientific and technological interest due to their high refractive indices, low melting temperatures, high dielectric constants and good infrared transmissions (Biswal *et al*., 1999).

Rare earth ions doped materials have broad band-region from ultraviolet to infrared, so these functional materials have been widely used in many fields, such as laser protection, nonlinear optical field, lights communication and laser materials. It has been ranked crucial elements by America, Japan etc., for developing high technology (Yaru *et al*, 2007). There are many candidates in the family viz., borate, chalcogenide, fluoride, germinate, oxynitride, silicate, phosphate, sulphide, zirconate, and tellurite glasses. Rare-earth ions, especially erbium, ytterbium and samarium, have played an important role in the development of broadband fiber amplifiers in optical communication technology during the past few decades (Sherief *et al*., 2004; Chun *et al*., 1999; and Weber *et al*., 1981). A rare-earth (RE) doped glass has been selected for its good performances i.e., melting at 1300°C, good chemical durability, low crystallization tendency. RE (lanthanide) ions are characterized and all of them have the same outer-shell configuration, namely $5s^2 5p^6 6s^2$, where the most stable ionization state is the trivalent one, with the 5s and 5p electrons remaining untouched and acting to screen the energy levels of the 4f electrons from the effect of the surrounding environment. The transition probabilities between 4f states, however, are sensitive to the ions surrounding the rare earth, and the design of a proper rare earth doped (RED) glass involves the study of a number of spectroscopic parameters (Quintas *et al*., 2007). Sm (Samarium) is a rare earth element and the facts that rare earth elements can work as oxygen scavengers have been well known for a long time; where Sm has a stronger affinity for oxygen than most transition metals, it is thus reasonably expected that Sm may act as an oxygen scavenger and help to suppress heterogeneous nucleations, and consequently improve the GFA (Sheng *et al*., 2005).

1.2 Problem Statement

Glasses containing large amounts of rare earth oxides have drawn great attention for use as nonlinear optical materials, display materials, fiber-optic communications and solid-state lasers for medical applications. However, the studies on samarium containing glasses have received relatively less attention than other lanthanide ions, despite many features of interest (Boonin *et al*., 2013). In previous years, there have been only few researches investigating in Sm_2O_3. Most of them focused on the other based glass former (silicate, phosphate, borate etc.) doped with Sm_2O_3. In particular, the Telluride borate based glasses system doped with the network modifier of Sm_2O_3 has rarely been reported in spite of its wide applications. Telluride is known to have a low melting point and flexibility which can form a glass when combined with alkaline metal ions, transition metal ions, and even rare earth ions (Mekki *et al*., 2005). Samarium ions were chosen in this study based on its potential ability as a network modifier and it possesses strong fluorescence intensity, rich energy levels, large emission cross section, high quantum efficiency and can work as oxygen scavengers. It is suitable to improve properties and develop new optical functions for glasses (Yaru *et al*., 2007). Hence, the relation between the position of Samarium ion in volatile TeO_2 – B_2O_3 glass network and the dielectric and optical properties of these glasses is expected to be quite interesting. The present study characterize Sm_2O_3 doped TeO_2-B_2O_3 glasses composition, physical, structural, optical and dielectric properties as a function of frequency and temperature as well as the effect of the rare earth ions (Sm^{3+} ions) introduced into these glasses systems. The present investigation on the dielectric properties is useful to understand the basic mechanism of the polarization process, and the parameters necessary for the basic understanding of the physical grounds of optical properties.

1.3 Objectives of the research

The present studies characterize B_2O_3 - TeO_2 - Sm_2O_3 glasses system composition with two different series. The investigation on the dielectric properties is useful to understand the mechanism of the polarization process. The objectives of this research are:

1. To prepare and synthesize the ternary glasses $(B_2O_3)_{30}$ $(Te0_2)_{70-x}$ $(Sm_2O_3)_x$ and $(B_2O_3)_{30-x}$ $(Te0_2)_{70}$ $(Sm_2O_3)_x$ (0.3 mol% ≥ x ≥ 1.2 mol%) by melt-quenched technique.
2. To investigate the effect of substitution of Sm_2O_3 as a network modifier on B_2O_3- TeO_2 based glass on the microstructure and to studying the optical and dielectric properties of $(B_2O_3)_{30}$ $(Te0_2)_{70-x}$ $(Sm_2O_3)_x$ and $(B_2O_3)_{30-x}$ $(Te0_2)_{70}$ $(Sm_2O_3)_x$ glasses system as a function of frequency ranging from 10^{-2} Hz to 10^6 Hz, at temperature ranging from 373 K to 493 K for different compositions of x.
3. To propose equivalent circuit model of the glasses samples at the various temperature and frequency.

1.4 Organization of the thesis

Chapter 1 shows the introduction of rare earth doped Tellurite borate glasses. Chapter 2 gives the previous research works carried out in the past and present literature of Tellurite based glass and the glass containing rare earth ions. Whereas Chapter 3 discusses the methodology including the basic and theories of physical, structural, optical and dielectric properties of the materials. The effect of rare earth ions doping both B_2O_3 - $Te0_2$ - Sm_2O_3 (BTS) glasses series on the physical, structural, optical and dielectric properties are analyzed and characterized in Chapter 4. Last but not least, the conclusion and the recommendation for future works are given in Chapter 5.

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