UNIVERSITI PUTRA MALAYSIA

ELASTIC PROPERTIES OF BOROTELLURITE GLASS DOPED WITH MANGANESE OXIDE

SYED PUTRA HAIZAM BIN SYED HASHIM

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ELASTIC PROPERTIES OF BOROTELLURITE GLASS DOPED WITH MANGANESE OXIDE

By

SYED PUTRA HAIZAM BIN SYED HASHIM

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DEDICATION

This thesis is dedicated to my very beloved father (Syed Hashim Bin Syed Endut), my caring mother (Saripah Norsidah Binti Syed Hussin), my father in law (Al Azmi Bin Ahmad), my mother in law (Nuriah Binti Mohd Noor), my lovely wife (Syazwani Binti Al Azmi) and my cute kids (Syed Muhammad El-Fatih and Syed Uwais Muhammad).

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

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May 2016

Chairman : Profesor Haji Sidek bin Haji Ab. Aziz, PhD
Faculty : Science

Now a day, glass had played very important role in our society. Various types of glasses with certain composition had been prepared for specific application. Even though preparation of glass basically for the purpose of application, in this research, only certain of the glass properties will be studied to analyse the tendency or the ability of prepared glass for further research in glass radiation sensor. Boro-tellurite glasses with the dopant of manganese ion were synthesis in this study by using melt and quench technique. Two series of $[(B_2O_3)_{0.3}(TeO_2)_{0.7}]_{(100-x)}$ $(MnO)_x$ with percentage of mole x from 0.1 to 0.4 glass had been successfully prepared to find the optimum percentage of amount of manganese ions needed so that the glass structure become fully amorphous. Then another four series of $[(B_2O_3)_{0.15}$ $(TeO_2)_{0.85}]_{(100-x)}(Mn_2O_3)_x$ with percentage of mole x from 1 to 5 glass had also successfully prepared. Samples prepared tested for X-ray diffraction technique. Result shows one sharp peak occurs for the percentage of mole at x = 0.1 and 0.2 for the first series of glass. However, the peak disappear at x= 0.3 and 0.4. For all other prepared samples, there were no peaks at all shows that the glasses have entered completely in amorphous structure. The densities of prepared sample were then determined by using the Archemedes's principle and the molar volume of the sample calculated. Structural bonding between the glasses molecules were analysed by using FT-IR spectrum. Then, by using ultrasonic MBS8000, longitudinal and transverse wave velocity had been determined. From the data of the density and the ultrasonic velocities, elastic properties of these glasses then obtained. Young's modulus, bulk's modulus and debye temperature of these borotellurite doped glass were found to be decrease with the increasing of manganese ions inside the glass network even after the glass were remelt. All series of glasses were found to have a decrease elastic modulus with temperature. All the trend were finally compared with Rocherulle's model and observed the same trend for all elastic velocities and elastic moduli. After remelting process, it was observed that the glass structure become more open and less strong.

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

PENCIRIAN KEKENYALAN KACA BOROTELLURITE TERDOP DENGAN MANGAN OKSIDA

Oleh

SYED PUTRA HAIZAM BIN SYED HASHIM

Mei 2016

Pengerusi : Profesor Haji Sidek bin Haji Ab. Aziz, PhD
Fakulti : Sains

Dini hari, kaca telah memainkan peranan yang amat penting dalam masyarakat kita. Pelbagai jenis kaca dengan komposisi yang berbeza telah disediakan untuk spesifikasi tertentu. Walaupun penyediaan kaca pada dasarnya bagi tujuan aplikasi, namun di dalam kajian ini, hanya beberapa ciri-ciri kaca akan dikaji bagi menganalisis kecenderungan atau keupayaan kaca yang disintesis untuk penyelidikan lanjut dalam sensor radiasi. Kaca Boro-tellurite dengan pendopan ion mangan di sintesis dalam kajian ini dengan menggunakan teknik lebur lindap. Dua siri $[(B_2O_3)_{0.3}(TeO_2)_{0.7}]_{(100-x)}(MnO)_x$ dengan peratusan mol x 0.1 – 0.4 kaca telah berjaya disentesis untuk mencari peratusan optimum jumlah ion mangan diperlukan supaya struktur kaca menjadi amorfus sepenuhnya. Kemudian empat siri yang seterusnya daripada $[(B_2O_3)_{0.15}(TeO_2)_{0.85}]_{(100-x)}(Mn_2O_3)_x$ dengan peratusan mol x 1-5 kaca telah berjaya disediakan. Sampel yang telah disediakan diuji dengan teknik pembelauan sinar-X (XRD). Keputusan menunjukkan satu puncak yang jelas berlaku untuk peratusan mol pada x = 0.1 dan 0.2 untuk siri pertama daripada kaca. Walau bagaimanapun, puncak hilang pada x = 0.3 dan 0.4. Untuk semua sampel lain, tidak ada puncak pada semua siri menunjukkan bahawa kaca telah pun berada sepenuhnya dalam struktur amorfus. Ketumpatan sampel-sampel kemudiannya diukur dengan menggunakan prinsip Archemedes dan isipadu molar sampel dikira. Ikatan struktur antara molekul kaca yang diperolehi seterusnya dianalisis dengan menggunakan spektrum FT-IR. Kemudian, dengan menggunakan MBS8000 ultrasonik, halaju gelombang membujur dan melintang ditentukan. Dari data ketumpatan dan halaju ultrasonik, sifat elastik cermin mata ini berjaya diperolehi. Modulus Young dan modulus Bulk bagi kaca borotellurite ini yang telah didopkan didapati berkurangan dengan peningkatan ion mangan di dalam rangkaian kaca walaupun selepas kaca telah dilebur semula. Modulus elastik pada semua siri kaca didapati menurun. Semua nilai penurunan graf akhirnya dibandingkan model Rocherulle dan dididapati penurunan yang sama untuk semua halaju anjal dan modulus elastik. Selepas proses peleburan semula, telah diperhatikan bahawa struktur kaca menjadi lebih terbuka dan kurang kuat.

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Finally, I wish to acknowledge the support of all my research group members and all distinguished figures who contributed to the success of my studies.

I certify that a Thesis Examination Committee has met on 19 May 2016 to conduct the final examination of Syed Putra Haizam bin Syed Hashim on his thesis entitled "Elastic Properties of Borotellurite Glass Doped with Manganese 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.

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Faculty of Science
Universiti Putra Malaysia
(Chairman)

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Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

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Associate Professor
Universiti Kebangsaan Malaysia
Malaysia
(External Examiner)

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

Date: 22 November 2016

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:

Sidek Bin HJ Abd Aziz, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Halim Bin Shaari, PhD
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Faculty of Science
Universiti Putra Malaysia
(Member)

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Faculty of Science
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Member of
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LIST OF ABBREVIATIONS

TEM	Transmission Electron Microscopy
SEM	Scanning Electron Microscopy
XRD	X-ray Diffractometry
FTIR	Fourier transform infrared
Wt	Total weight of chemcals
cm	Centimeter
mm	Millimeter
nm	Nanometer
μm	Mircometer
mL	Milliliter
mV	Millivolt
^{o}C	Degree Celsius
LLP	Long lasting phosphorescence

CHAPTER 1

INTRODUCTION

1.1 Research background

Glass had been very common in our everyday lives. Until today, glass had been practiced for many roles and applications due to their physical figure and properties. In radiation field, previously, borate ceramic was used as thermo luminescent dosimeter. Nevertheless, since 2011 glass was introduced as a dosimeter due to their photo luminescent properties (David *et al*, 2011).

Characteristics or properties of these glasses mainly are due to the chemical composition and the structure of the glass former, modifier or intermediate. Stanworth (1950) built open the models of Zachariasen, Smekal and Pauling and proposed that cations could be separated into three different roles: network formers, modifiers and intermediate species.

The study of tellurite glasses is of scientific and technical interest because they have low melting points, high refractive index, high dielectric constant and good infrared transmission (Thomas *et al*, 2012). Tellurium oxide (TeO_2) is a conditional glass former and forms glass only with a modifier such as alkali, alkaline earth and transition metal oxides or other glass formers. In a binary tellurite glass, the basic structural unit of TeO_4 is a trigonalbipyramid (tbp) with a lone pair of electrons and the structural units take the Te-O-Te bond for glass formation (Mekki *et al*, 2009).

Halimah (2005) had synthesized pure borotellurite glass which tellurite as a glass former and borate as a modifier. In her research the density and elastic properties of this glass was studied. She was found that the density of the glass was increased gradually with the increase of modifiers.

1.2 Problem statement

A spirited debate occurred in the literature concerning the effect of addition of transition elements into the borate and tellurite glass systems, however the addition of manganese into the borotellurite glass system was limited with no definitive answers to the fundamental topics. Although many properties of tellurite glass such as low melting point, high chemical durability and high refractive index have attracted a number of researchers because of their wide-ranging industrial and technical applications, no systematic study of physical, structure and elastic properties of borotellurite glass with the dopant of manganese has been reported, including the effect of remelting of these glasses. Therefore, a detailed investigation

of the physical, structural and elastic properties of borotellurite glass with the addition of manganese oxide will be carried out and the results of this study are presented in this thesis.

1.3 Objectives of the research

This study was carried out based on some clear and precise objectives. The purposes of this research are:

1. to synthesize $[(B_2O_3)_{0.15}(TeO_2)_{0.85}]_{(100-x)}(MnO)_x$ and $[(B_2O_3)_{0.15}(TeO_2)_{0.85}]_{(100-x)}(Mn_2O_3)_x$ glasses.
2. to study the effect and influence of MnO and Mn_2O_3 on the structure, physical and elastic properties of boro-tellurite glass.
3. to compare the theoretical values based on Rocherulle's model of MnO and Mn_2O_3 doped glasses.
4. to investigate the effect of remelting on the glass matrix from physical, structural and elastic properties.

1.4 Scope of the study

In order to achieve the objective of the study, the glass samples based on the stiochiometric equation of $[(B_2O_3)_{0.15}(TeO_2)_{0.85}]_{(100-x)}(MnO)_x$ and $[(B_2O_3)_{0.15}(TeO_2)_{0.85}]_{(100-x)}(Mn_2O_3)_x$ where *x* between 0.1 to 5 mol% will be prepared by mixing B_2O_3, TeO_2, MnO and Mn_2O_3 powders using conventional melting and quenching technique. The binary boro-tellurite glass samples will also be prepared to serve as a reference material.

The structure of $[(B_2O_3)_{0.15}(TeO_2)_{0.85}]_{(100-x)}(MnO)_x$ and $[(B_2O_3)_{0.15}(TeO_2)_{0.85}]_{(100-x)}(Mn_2O_3)_x$ glasses will be measured using X-ray diffraction technique to confirm the amorphocity of the glass sample. In order to investigate the structural properties of the glass samples, density measurement using Archimedes' technique was done. Then molar volume of all prepared samples, was then calculated.

The ultrasonic wave velocity of the $[(B_2O_3)_{0.15}(TeO_2)_{0.85}]_{(100-x)}(MnO)_x$ and $[(B_2O_3)_{0.15}(TeO_2)_{0.85}]_{(100-x)}(Mn_2O_3)_x$ glasses will be measured using Matec DSP MBS 8000. The values of the wave velocities were then used to measure the elastic properties of the glasses. From these values, physical properties or the strength of the glass can be known and determined.

1.5 Important of the study

Manganese doped glass technologically important because of its photoluminescence properties which is very useful for radiation glass detector technology (Amany A.E., 2012). Besides that, the glass microstructure can predict the optical and electrical properties. The structure measurement also can interpret the glass physical and chemical properties.

In this research, MnO and Mn_2O_3 had been chosen as an oxide to be doped into the borotellurite glass because of their good properties to improve the glass quality by increasing mechanical properties and enhancing chemical durability.

1.6 Chapters organization

The thesis is structured in five chapters, conclusions and references. In chapter 1 the general introduction regarding general explanation of glass and the aim of the overall research is well presented.

Chapter 2 presents the literature review on glass properties in general which include characterization used in the analyses of vitreous structure, i.e. X-ray diffraction, density, molar volume, FT-IR spectra, ultrasonic testing and Rocherull's theoretical model calculation.

Chapter 3 clarifies all the series codes and describes the melt and quenching method used to obtain tellurite materials studied in this work as well as the experimental measurement systems used throughout the research.

Chapters 4 present all results obtained in the studies of all series of borotellurite systems doped with manganese ions by melting and quenching technique method including detailed discussions on each characterization.

Chapter 5 will conclude the overall outcomes of this work.

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