



UNIVERSITI PUTRA MALAYSIA

***EFFECT OF GAMMA RADIATION ON ELASTIC AND OPTICAL
PROPERTIES OF Tm₂O₃/CeO₂-DOPED ZINC BOROTELLURITE GLASS
SYSTEM***

HASNIMULYATI BINTI LAODING

FS 2018 7



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SYSTEM**

By

HASNIMULYATI BINTI LAODING

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

November 2017

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*This thesis is specially dedicated to my mother, Bungatang Bte Petalimpe,
my lecturers, family and friends.*

Thank you for encouraging me to finish this journey.



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

EFFECT OF GAMMA RADIATION ON ELASTIC AND OPTICAL PROPERTIES OF Tm₂O₃/CeO₂-DOPED ZINC BOROTELLURITE GLASS SYSTEM

By

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November 2017

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

Two series of zinc borotellurite glasses that were doped with thulium and cerium oxide were successfully fabricated using the known melt-quenching technique. The glasses were prepared based on the empirical formula of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$ with the concentration of the rare earth oxides were varied from 0.00 to 0.05 mol. The structural, elastic and optical properties of the glass samples were tested using densimeter, Fourier Transform Infrared Spectrometer (FTIR), X-ray diffraction (XRD) analysis, ultrasound technique and UV-Visible Spectrophotometer (UV-VIS). The samples with 1 mol% of dopants were exposed to gamma radiation with dose ranging from 10 to 35 kGy and all the tests were done before and after the radiation process. As the rare earth oxides were added into the glass system, the density of the glass was found to increase from 3.69 to 4.99 g/cm³. Meanwhile, the molar volume (V_m) of glass decreases rapidly at 0.01 mol of the additional oxide and varies beyond that. The minimum and maximum values for V_m are 25.57 and 31.75 cm³/mol respectively. In terms of FTIR spectra, the absence of Zn-O, Tm-O and Ce-O bonds implies that these bonds have been broken and these oxides take the role as network modifier by filling up the interstitial spaces inside the glass network. The amorphous nature of the glass is confirmed using XRD analysis. Besides, the elastic moduli and other elastic parameters of the glass under study generally found to increase with the substitution of Tm₂O₃ and CeO₂ while the value of Poisson's ratio lies in the range of 0.2633 to 0.2740. In terms of optical parameters, all of them exhibit some variations as the dopants are added. After the glass samples with 1 mol% of rare earth oxides is radiated with gamma rays, the density for Ce-doped glass found to decrease from 4.600 to 3.576 g/cm³ whereas for Tm-doped glass, it varies with minimum value of 4.57 g/cm³ and maximum value of 4.66 g/cm³. Meanwhile, the molar volume for both glass series is totally opposite to density. The glass samples also maintain their amorphous nature after radiation even though new absorption bands were found to be produce in the FTIR analysis. The elastic moduli for cerium doped glass show a decrement after exposed to gamma rays while in thulium doped glass, they show an

increasing trend. Furthermore, the optical band gaps for both glass series tend to decrease with the increment of radiation dose. In conclusion, the addition of thulium and cerium oxide into zinc borotellurite glass found to alter the glasses' properties differently. In addition, the effects of gamma radiation also are different for both glass series. This research can contribute new knowledge regarding rare-earth doped zinc borotellurite glass and the effect of gamma radiation on them. Besides that, these glasses also have a high potential to be used as gain media in laser devices for medical surgery and as fibre amplifier in radiation-exposed environment.



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

**KESAN SINAR GAMA KEPADA SIFAT KENYAL DAN OPTIK SISTEM
KACA Tm_2O_3/CeO_2 DIDOP DENGAN ZINK BOROTELURIT**

Oleh

HASNIMULYATI BINTI LAODING

November 2017

Pengerusi : Profesor Madya Halimah Mohamed Kamari, PhD
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Dua siri kaca zink borotelurit yang didop dengan tulium dan serium oksida berjaya dihasilkan menggunakan teknik sepuh lindap yang terkenal. Kaca ini disediakan berdasarkan formula empirikal $\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.7}[ZnO]_{0.3}\}_{1-x}\{Tm_2O_3/CeO_2\}_x$ dengan kepekatan nadir bumi oksida divariasikan daripada 0.00 hingga 0.05 mol. Sifat struktur, kenyal dan optik sampel kaca diuji menggunakan densimeter, Spektrometer Inframerah Jelmaan Fourier (FTIR), analisis pembelauan sinar-X (XRD), teknik ultrabunyi dan Spektrofotometer UV-nampak (UV-VIS). Selepas itu, sampel dengan 1 mol% dopan didedahkan kepada sinar gama dengan julat dos dari 10 ke 35 kGy dan semua ujian dilakukan sebelum dan selepas proses radiasi. Apabila nadir bumi oksida ini ditambahkan ke dalam sistem kaca, ketumpatan kaca itu didapati meningkat dari 3.69 kepada 4.99 g/cm³. Sementara itu, isipadu molar kaca (V_m) turun secara mendadak pada 0.01 mol oksida tambahan dan bervariasi selepas itu. Nilai minima dan maksima untuk V_m masing-masing adalah 25.57 dan 31.75 cm³/mol. Dari segi spektra FTIR, ketidakhadiran ikatan Zn-O, Tm-O dan Ce-O bererti bahawa ikatan-ikatan ini telah terputus dan bahan oksida ini mengambil peranan sebagai pengubah rangkaian dengan memenuhi ruang interstis di dalam rangkaian kaca itu. Sifat amorfus kaca itu dipastikan dengan menggunakan analisis XRD. Selain itu, modulus kenyal dan parameter kenyal yang lain untuk kaca yang dipelajari ini secara umumnya didapati meningkat dengan penggantian Tm_2O_3 dan CeO_2 manakala nilai nisbah Poisson terletak pada julat 0.2633 hingga 0.2740. Dari segi parameter optik, kesemuanya menunjukkan perubahan apabila dopan ditambah. Selepas sampel kaca dengan 1 mol% nadir bumi diradiasi dengan sinar gama, ketumpatan untuk kaca yang didop Ce didapati menurun daripada 4.600 kepada 3.576 g/cm³ sedangkan untuk kaca yang didop Tm, ianya berubah dengan nilai minima 4.57 g/cm³ dan nilai maksima 4.66 g/cm³. Sementara itu, isipadu molar untuk kedua-dua siri kaca adalah bertentangan dengan ketumpatan. Sampel kaca ini juga mengekalkan sifat amorfus mereka selepas radiasi walaupun jalur penyerapan baru ditemui terhasil di dalam analisis FTIR. Modulus-modulus kenyal untuk kaca yang didop dengan serium menunjukkan penurunan selepas didedahkan kepada sinar gama manakala di dalam kaca yang didop dengan tulium, mereka menunjukkan trend

yang meningkat. Tambahan pula, nilai jurang jalur optik bagi kedua-dua siri cenderung untuk menurun dengan kenaikan dos radiasi. Kesimpulannya, penambahan tulium dan serium oksida kepada kaca zink borotelurit telah merubah ciri-ciri kaca tersebut secara berbeza-beza. Selain itu, kesan sinar gama juga berbeza bagi kedua-dua siri kaca tersebut. Kajian ini boleh menyumbang ilmu baharu mengenai kaca zink borotelurit yang didop dengan nadir bumi dan kesan sinar gama kepada kaca-kaca tersebut. Selain itu, kaca-kaca ini juga mempunyai potensi yang tinggi untuk digunakan sebagai media gandaan di dalam peralatan laser untuk pembedahan perubatan dan sebagai amplifiler gentian dalam persekitaran yang terdedah pada sinar gama.



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I certify that a Thesis Examination Committee has met on 9 November 2017 to conduct the final examination of Hasnimulyati binti Laoding on her thesis entitled "Effect of Gamma Radiation on Elastic and Optical Properties of Tm_2O_3/CeO_2 -Doped Zinc Borotellurite Glass System" 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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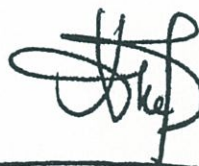
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TABLE OF CONTENTS

	Page
ABSTRACT	i
ABSTRAK	iii
ACKNOWLEDGEMENTS	v
APPROVAL	vi
DECLARATION	viii
LIST OF TABLES	xiii
LIST OF FIGURES	xvi
LIST OF ABBREVIATIONS	xxii
CHAPTER	
1 INTRODUCTION	1.1
1.1 Research background	1.1
1.1.1 Definition of glass	1.1
1.1.2 Tellurite glass (TeO_2)	1.2
1.1.3 Borate (B_2O_3)	1.3
1.1.4 Zinc oxide (ZnO)	1.3
1.1.5 Thulium oxide (Tm_2O_3)	1.4
1.1.6 Cerium oxide (CeO_2)	1.4
1.2 Problem Statement	1.5
1.3 Objectives of study	1.6
1.4 Hypotheses	1.6
1.5 Scope and limitations	1.7
1.6 Significance of study	1.7
2 LITERATURE REVIEW	2.1
2.1 Structural and physical properties	2.1
2.2 Elastic properties	2.3
2.3 Optical properties	2.7
3 THEORY	3.1
3.1 Formation of glass	3.1
3.2 Structure of tellurite glass network	3.8
3.3 Structure of borate glass network	3.10
3.4 Elastic properties of glass	3.12
3.4.1 Study of elasticity	3.12
3.4.2 Bulk compression model	3.14
3.4.3 Ring deformation model	3.16
3.4.4 Makishima-Mackenzie model	3.17
3.4.5 Rocherulle model	3.18
3.5 Optical properties of glass	3.20
3.6 Effect of gamma radiation on glass	3.24

4	METHODOLOGY	4.1
	4.1 Sample preparation	4.1
	4.2 Sample characterization	4.4
	4.2.1 Density and molar volume measurement	4.4
	4.2.2 Fourier Transform Infrared Spectroscopy (FTIR)	4.5
	4.2.3 X-ray Diffraction (XRD)	4.5
	4.2.4 Ultrasound measurement	4.6
	4.2.5 UV-Visible Spectroscopy	4.7
	4.2.6 Gamma radiation machine	4.8
5	RESULT AND DISCUSSION	5.1
	5.1 Physical and structural properties before radiation process	5.1
	5.1.1 FTIR analysis	5.1
	5.1.2 Density and molar volume	5.7
	5.1.3 XRD analysis	5.9
	5.2 Elastic properties before radiation process	5.11
	5.2.1 Ultrasonic velocity	5.11
	5.2.2 Elastic moduli	5.15
	5.2.3 Poisson's ratio	5.20
	5.2.4 Microhardness	5.21
	5.2.5 Debye temperature	5.23
	5.2.6 Acoustic impedance	5.25
	5.2.7 Softening temperature	5.27
	5.2.8 Elastic theoretical models	5.29
	5.3 Optical properties before radiation process	5.45
	5.3.1 Optical band gap	5.45
	5.3.2 Urbach energy	5.51
	5.3.3 Refractive index, molar refraction and polarizability	5.54
	5.3.4 Optical basicity	5.59
	5.3.5 Metallization criterion	5.61
	5.4 Physical and structural properties after radiation process	5.62
	5.4.1 FTIR analysis	5.62
	5.4.2 Density and molar volume	5.69
	5.4.3 XRD analysis	5.71
	5.5 Elastic properties after radiation process	5.73
	5.5.1 Ultrasonic velocity	5.73
	5.5.2 Elastic moduli	5.75
	5.5.3 Poisson's ratio	5.80
	5.5.4 Microhardness	5.81
	5.5.5 Debye temperature	5.83
	5.5.6 Acoustic impedance	5.84
	5.5.7 Softening temperature	5.86
	5.3 Optical properties after radiation process	5.87
	5.3.1 Optical band gap	5.87

5.3.2	Urbach energy	5.93
5.3.3	Refractive index, molar refraction and polarizability	5.96
5.3.4	Optical basicity	5.99
5.3.5	Metallization criterion	5.101
6	CONCLUSIONS AND FUTURE RESEARCH	6.1
6.1	Conclusions	6.1
6.2	Future research	6.3
	REFERENCES	A-1
	BIODATA OF STUDENT	B-1
	LIST OF PUBLICATIONS	C-1



LIST OF TABLES

Table		Page
5.1	Band center (B), band area (A) and assignments for $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3\}_x$	5.4
5.2	Band center (B), band area (A) and assignments for $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{CeO}_2\}_x$	5.5
5.3	Density and molar volume of thulium and cerium doped zinc borotellurite glass.	5.7
5.4	Longitudinal (V_L) and shear velocity (V_S) of thulium and cerium doped zinc borotellurite glass.	5.12
5.5	Elastic moduli, Poisson's ratio, microhardness, Debye temperature, acoustic impedance, softening temperature and fractal bond connectivity of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3\}_x$	5.15
5.6	Elastic moduli, Poisson's ratio, microhardness, Debye temperature, acoustic impedance, softening temperature and fractal bond connectivity of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{CeO}_2\}_x$	5.16
5.7	First-order stretching force constant (f), number of cross-link per unit cation (n_c), number of cations per unit glass formula unit (N_c), coordination number (n) and bond length (r) for each constituent oxides.	5.29
5.8	Total number of cations per unit glass formula unit (η), average cross-link density per unit formula ($\langle n_c \rangle$), average stretching force constant (F), and number of bond per unit volume (n_b) of thulium-doped zinc borotellurite glass.	5.30
5.9	Total number of cations per unit glass formula unit (η), average cross-link density per unit formula ($\langle n_c \rangle$), average stretching force constant (F), and number of bond per unit volume (n_b) of cerium-doped zinc borotellurite glass.	5.30
5.10	Elastic moduli, Poisson's ratio (σ_{bc}), ratio K_{bc}/K_e and atomic ring size (ℓ) of thulium-doped zinc borotellurite glass by bulk compression model.	5.31
5.11	Elastic moduli, Poisson's ratio (σ_{bc}), ratio K_{bc}/K_e and atomic ring size (ℓ) of cerium-doped zinc borotellurite glass by bulk compression model.	5.31
5.12	Packing density and dissociation energy of all the chemical oxides used in the study.	5.36

5.13	Total packing density (V_t), total dissociation energy (G_t), bulk modulus (K_{mm}), shear modulus (G_{mm}), Young's modulus (E_{mm}) and Poisson's ratio (σ_{mm}) of thulium-doped zinc borotellurite glass.	5.36
5.14	Total packing density (V_t), total dissociation energy (G_t), bulk modulus (K_{mm}), shear modulus (G_{mm}), Young's modulus (E_{mm}) and Poisson's ratio (σ_{mm}) of cerium-doped zinc borotellurite glass.	5.37
5.15	Packing density (C_t), bulk modulus (K_r), shear modulus (G_r), Young's modulus (E_r) and Poisson's ratio of thulium-doped zinc borotellurite glass	5.39
5.16	Packing density (C_t), bulk modulus (K_r), shear modulus (G_r), Young's modulus (E_r) and Poisson's ratio of cerium-doped zinc borotellurite glass.	5.39
5.17	Electronic transitions in thulium and cerium-doped zinc borotellurite glass.	5.45
5.18	Direct and indirect optical band gap of thulium and cerium-doped zinc borotellurite glass.	5.48
5.19	Urbach energies of thulium and cerium-doped zinc borotellurite glass.	5.53
5.20	Refractive index (n), molar refraction (R_m), molar (α_m) and oxide ion polarizability ($\alpha_{O^{2-}}$) of thulium and cerium-doped zinc borotellurite glass.	5.55
5.21	Optical basicity of thulium and cerium-doped zinc borotellurite glass.	5.59
5.22	Metallization criterion of thulium and cerium-doped zinc borotellurite glass.	5.61
5.23	Band center (B), band area (A) and assignments for thulium doped zinc borotellurite glass after radiation process.	5.64
5.24	Assignments of infrared absorption band in thulium and cerium doped zinc borotellurite glass after radiation process.	5.65
5.25	Band center (B), band area (A) and assignments for cerium doped zinc borotellurite glass after radiation process.	5.66
5.26	Density and molar volume of thulium and cerium-doped glass after radiation process.	5.69
5.27	Longitudinal velocity (v_L), shear velocity (v_S) and atomic ring size (ℓ) of thulium-doped zinc borotellurite glass before and after radiation process.	5.73

5.28	Longitudinal velocity (v_L), shear velocity (v_S) and atomic ring size (ℓ) of cerium-doped zinc borotellurite glass before and after radiation process.	5.74
5.29	Elastic moduli, Poisson's ratio, microhardness, Debye temperature, acoustic impedance, softening temperature and fractal bond connectivity of thulium-doped zinc borotellurite glass before and after radiation process.	5.76
5.30	Elastic moduli, Poisson's ratio, microhardness, Debye temperature, acoustic impedance, softening temperature, fractal bond connectivity of cerium-doped zinc borotellurite glass before and after radiation process.	5.77
5.31	Direct and indirect optical band gap of thulium and cerium-doped zinc borotellurite glass before and after radiation process.	5.91
5.32	Urbach energy (ΔE) of thulium and cerium-doped zinc borotellurite glass before and after radiation process.	5.94
5.33	Refractive index (n), molar refraction (R_m), molar (α_m) and oxide ion polarizability ($\alpha_{O^{2-}}$) of thulium and cerium-doped zinc borotellurite glass before and after radiation process.	5.96
5.34	Optical basicity of thulium and cerium-doped zinc borotellurite glass before and after radiation process.	5.100
5.35	Metallization criterion of thulium and cerium-doped glass before and after radiation process.	5.101

LIST OF FIGURES

Figure		Page
1.1	Network structure of crystalline and amorphous solid	1.2
3.1	Growth and nucleation rate for poor glass former	3.4
3.2	Growth and nucleation rate for good glass former	3.5
3.3	A Time-Temperature-Transformation curve	3.6
3.4	Schematic plot of glass transition	3.6
3.5	TeO ₂ unit in paratellurite α -TeO ₂	3.8
3.6	Structural units of tellurite systems	3.9
3.7	Transformation of tellurite structural units	3.9
3.8	Transformation of BO ₃ unit to BO _{2/2} O ⁻ M ⁺ unit accompanied with NBO	3.10
3.9	Conversion from BO ₃ into BO ₄ group	3.10
3.10a	Boroxol group	3.11
3.10b	Pentaborate group	3.11
3.10c	Triborate group	3.11
3.10d	Diborate group	3.11
3.11a	Metaborate group	3.12
3.11b	Orthoborate group	3.12
3.11c	Pyroborate group	3.12
3.11d	Tetraborate group	3.12
3.12a	Illustration diagram of elastic constants : Young's modulus	3.12
3.12b	Illustration diagram of elastic constants : Shear modulus	3.12
3.12c	Illustration diagram of elastic constants : Bulk modulus	3.12
3.13	Bond compression in three-dimensional network as glass is subjected to isotropic deformation	3.16
3.14	Bond compression and bending in layered structure as glass is	3.16

	subjected to isotropic deformation	
3.15	Bond compression and bending in chain structure as glass is subjected to isotropic deformation	3.16
3.16	Ring deformation model; $l = (\text{number of bond} \times \text{bond length}) / 2\pi$	3.17
3.17	Illustration of the variation of cross-link density in network structure	3.19
3.18	Direct allowed transition	3.21
3.19	Indirect allowed transition	3.22
3.20	Photoelectric effect that occurs after radiation process	3.24
3.21	Compton scattering that occur after radiation process	3.25
3.22	Electron positron pair production that occur after radiation process	3.25
4.1	Preparation process for $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$ glasses	4.2
4.2	Flow process of glass preparation	4.3
4.3	Characterizations and related properties	4.4
4.4	X-ray scattering in material	4.5
5.1	FTIR spectra of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3\}_x$	5.2
5.2	FTIR spectra of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{CeO}_2\}_x$	5.3
5.3	Deconvolution of FTIR spectra for $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.09}\{\text{Tm}_2\text{O}_3\}_{0.01}$	5.3
5.4	Band area of TeO_3 and TeO_4 structural units of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3\}_x$ glass system	5.4
5.5	Band area of BO_3 and BO_4 structural units of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3\}_x$ glass system	5.5
5.6	Band area of TeO_3 and TeO_4 structural units of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{CeO}_2\}_x$ glass system	5.6
5.7	Band area of BO_3 and BO_4 structural units of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{CeO}_2\}_x$ glass system	5.6

5.8	Density and molar volume of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$	5.7
5.9	Relation between density and total molecular weight of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$ glass system	5.8
5.10	XRD spectra of thulium-doped zinc borotellurite glass	5.10
5.11	XRD spectra of cerium-doped zinc borotellurite glass	5.10
5.12	XRD spectra of zinc borotellurite glass with 1 mol% dopant concentration.	5.11
5.13	Variation of longitudinal and shear velocity of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$ glass system	5.12
5.14	Relation between density and ultrasonic velocities of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3\}_x$ glass system	5.13
5.15	Relation between density and ultrasonic velocities of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{CeO}_2\}_x$ glass system	5.13
5.16	Longitudinal and shear modulus of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3\}_x$ glass system	5.17
5.17	Bulk and Young's modulus of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3\}_x$ glass system	5.17
5.18	Longitudinal and shear modulus of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{CeO}_2\}_x$ glass system	5.18
5.19	Bulk and Young's modulus of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{CeO}_2\}_x$ glass system	5.18
5.20	Relation between longitudinal modulus, L, and packing density, V_t , of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$ glass system	5.19
5.21	Variation of Poisson's ratio of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$ glass system	5.20
5.22	Relation between Poisson's ratio, σ , and fractal bond connectivity, d, of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$ glass system	5.21
5.23	Microhardness of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$ glass system	5.22

5.24	Relation between microhardness, H, and packing density, V_t , of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$ glass system	5.23
5.25	Debye temperature of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$ glass system	5.24
5.26	Mean velocity of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$ glass system	5.25
5.27	Acoustic impedance of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$ glass system	5.26
5.28	Relation between acoustic impedance, Z, and number of cross-link density per unit volume, $\langle n_c \rangle$ of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$ glass system	5.26
5.29	Softening temperature $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$	5.27
5.30	Relation between softening temperature, T_s , and microhardness, H, of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$ glass system	5.28
5.31	Elastic moduli of thulium-doped zinc borotellurite glass system by bulk compression model	5.32
5.32	Elastic moduli of cerium-doped zinc borotellurite glass system by bulk compression model	5.32
5.33	The ratio of K_{bc}/K_e of thulium and cerium-doped zinc borotellurite glass	5.34
5.34	Atomic ring size of thulium and cerium-doped zinc borotellurite glass	5.35
5.35	Elastic moduli of thulium-doped zinc borotellurite glass by Makishima-Mackenzie model	5.38
5.36	Elastic moduli of cerium-doped zinc borotellurite glass by Makishima-Mackenzie model	5.38
5.37	Variation of packing density, C_t of thulium and cerium-doped zinc borotellurite glass	5.40
5.38	Comparison of theoretical and experimental Young's modulus, E, of thulium-doped zinc borotellurite glass system	5.41
5.39	Comparison of theoretical and experimental bulk modulus, K, of thulium-doped zinc borotellurite glass system	5.41

5.40	Comparison of theoretical and experimental shear modulus, G , of thulium-doped zinc borotellurite glass system	5.42
5.41	Comparison of theoretical and experimental Poisson's ratio, σ , of thulium-doped zinc borotellurite glass system	5.42
5.42	Comparison of theoretical and experimental Young's modulus, E , of cerium-doped zinc borotellurite glass system	5.43
5.43	Comparison of theoretical and experimental bulk modulus, K , of cerium-doped zinc borotellurite glass system	5.43
5.44	Comparison of theoretical and experimental shear modulus, G , of cerium-doped zinc borotellurite glass system	5.44
5.45	Comparison of theoretical and experimental Poisson's ratio, σ , of cerium-doped zinc borotellurite glass system	5.44
5.46	Absorbance spectra of thulium-doped zinc borotellurite glass	5.45
5.47	Absorbance spectra of cerium-doped zinc borotellurite glass	5.46
5.48	Plot of $(\alpha\hbar\omega)^2$ vs $\hbar\omega$ for thulium-doped zinc borotellurite glass	5.46
5.49	Plot of $(\alpha\hbar\omega)^{1/2}$ vs $\hbar\omega$ for thulium-doped zinc borotellurite glass	5.47
5.50	Plot of $(\alpha\hbar\omega)^2$ vs $\hbar\omega$ for cerium-doped zinc borotellurite glass	5.47
5.51	Plot of $(\alpha\hbar\omega)^{1/2}$ vs $\hbar\omega$ for cerium-doped zinc borotellurite glass	5.48
5.52	Direct and indirect optical band gap of thulium-doped zinc borotellurite glass	5.49
5.53	Direct and indirect optical band gap of cerium-doped zinc borotellurite glass	5.50
5.54	Comparison of optical band gap of thulium and cerium-doped zinc borotellurite glass	5.51
5.55	Plot of $\ln \alpha$ vs $\hbar\omega$ of thulium-doped zinc borotellurite glass	5.52
5.56	Plot of $\ln \alpha$ vs $\hbar\omega$ of cerium-doped zinc borotellurite glass	5.52
5.57	Variation of Urbach energies for thulium and cerium-doped zinc borotellurite glass	5.53
5.58	Variation of refractive index and molar refraction of thulium-doped zinc borotellurite glass	5.54
5.59	Molar and oxide ion polarizability of thulium-doped zinc	5.56

	borotellurite glass	
5.60	Variation of refractive index and molar refraction of cerium-doped zinc borotellurite glass	5.56
5.61	Variation of molar and oxide ion polarizability of cerium-doped zinc borotellurite glass	5.57
5.62	Refractive index of thulium and cerium-doped zinc borotellurite glass	5.58
5.63	Optical basicity of thulium and cerium-doped zinc borotellurite glass	5.59
5.64	Variation of metallization criterion of thulium and cerium-doped zinc borotellurite glass	5.61
5.65	FTIR spectra of thulium doped zinc borotellurite glass after radiation process	5.63
5.66	FTIR spectra of cerium doped zinc borotellurite glass after radiation process	5.63
5.67	Band area of TeO_3 and TeO_4 structural units for $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3\}_{0.01}$ glass system after radiation process	5.64
5.68	Band area of BO_3 and BO_4 structural units for $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3\}_{0.01}$ glass system after radiation process	5.65
5.69	Band area of TeO_3 and TeO_4 structural units for $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{CeO}_2\}_{0.01}$ glass system after radiation process	5.67
5.70	Band area of BO_3 and BO_4 structural units for $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{CeO}_2\}_{0.01}$ glass system after radiation process	5.67
5.71	Variation of density and molar volume of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3\}_{0.01}$ glass system after radiation process	5.69
5.72	Variation of density and molar volume of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{CeO}_2\}_{0.01}$ glass system after radiation process	5.70
5.73	XRD spectra of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3\}_{0.01}$ glass system after radiation process	5.71

5.74	XRD spectra of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{CeO}_2\}_{0.01}$ glass system after radiation process	5.71
5.75	Comparison of XRD spectra of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_{0.01}$ glass system before and after radiation	5.72
5.76	Variation of longitudinal (v_L) and shear velocity (v_S) of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3\}_{0.01}$ glass system before and after radiation process	5.74
5.77	Variation of longitudinal (v_L) and shear velocity (v_S) of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{CeO}_2\}_{0.01}$ glass system before and after radiation process	5.75
5.78	Longitudinal and shear modulus of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3\}_{0.01}$ glass system before and after radiation process	5.75
5.79	Bulk and Young's modulus of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3\}_{0.01}$ glass system before and after radiation process	5.78
5.80	Longitudinal and shear modulus of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{CeO}_2\}_{0.01}$ glass system before and after radiation process	5.78
5.81	Bulk and Young's modulus of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{CeO}_2\}_{0.01}$ glass system before and after radiation process	5.79
5.82	Poisson's ratio of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_{0.01}$ glass system before and after radiation process	5.80
5.83	Fractal bond connectivity (d) of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_{0.01}$ glass system before and after radiation process	5.81
5.84	Microhardness of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_{0.01}$ glass system before and after radiation process	5.82
5.85	Debye temperature of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_{0.01}$ glass system before and after radiation process	5.83
5.86	Acoustic impedance $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_{0.01}$ glass system before and after radiation process	5.85

5.87	Variation of softening temperature of {[(TeO ₂) _{0.7} (B ₂ O ₃) _{0.3}] _{0.7} [ZnO] _{0.3} } _{0.99} {Tm ₂ O ₃ /CeO ₂ } _{0.01} glass system before and after radiation process	5.87
5.88	Absorption spectra of thulium-doped zinc borotellurite glass before and after radiation process	5.88
5.89	Absorption spectra of cerium-doped zinc borotellurite glass before and after radiation process	5.88
5.90	Plot of $(\alpha\hbar\omega)^2$ vs $\hbar\omega$ of {[(TeO ₂) _{0.7} (B ₂ O ₃) _{0.3}] _{0.7} [ZnO] _{0.3} } _{0.99} {Tm ₂ O ₃ } _{0.01} glass system after radiation process	5.89
5.91	Plot of $(\alpha\hbar\omega)^{1/2}$ vs $\hbar\omega$ of {[(TeO ₂) _{0.7} (B ₂ O ₃) _{0.3}] _{0.7} [ZnO] _{0.3} } _{0.99} {Tm ₂ O ₃ } _{0.01} glass system after radiation process	5.89
5.92	Plot of $(\alpha\hbar\omega)^2$ vs $\hbar\omega$ of {[(TeO ₂) _{0.7} (B ₂ O ₃) _{0.3}] _{0.7} [ZnO] _{0.3} } _{0.99} {CeO ₂ } _{0.01} glass system after radiation process	5.90
5.93	Plot of $(\alpha\hbar\omega)^{1/2}$ vs $\hbar\omega$ of {[(TeO ₂) _{0.7} (B ₂ O ₃) _{0.3}] _{0.7} [ZnO] _{0.3} } _{0.99} {CeO ₂ } _{0.01} glass system after radiation process	5.90
5.94	Variation of direct and indirect optical band gap of {[(TeO ₂) _{0.7} (B ₂ O ₃) _{0.3}] _{0.7} [ZnO] _{0.3} } _{0.99} {Tm ₂ O ₃ } _{0.01} glass system before and after radiation process	5.91
5.95	Variation of direct and indirect optical band gap of {[(TeO ₂) _{0.7} (B ₂ O ₃) _{0.3}] _{0.7} [ZnO] _{0.3} } _{0.99} {CeO ₂ } _{0.01} glass system before and after radiation process	5.92
5.96	Plot of $\ln \alpha$ vs $\hbar\omega$ of {[(TeO ₂) _{0.7} (B ₂ O ₃) _{0.3}] _{0.7} [ZnO] _{0.3} } _{0.99} {Tm ₂ O ₃ } _{0.01} glass system after radiation process	5.93
5.97	Plot of $\ln \alpha$ vs $\hbar\omega$ of {[(TeO ₂) _{0.7} (B ₂ O ₃) _{0.3}] _{0.7} [ZnO] _{0.3} } _{0.99} {CeO ₂ } _{0.01} glass system after radiation process	5.94
5.98	Variation of Urbach energy for {[(TeO ₂) _{0.7} (B ₂ O ₃) _{0.3}] _{0.7} [ZnO] _{0.3} } _{0.99} {Tm ₂ O ₃ /CeO ₂ } _{0.01} glass system before and after radiation process	5.95
5.99	Variation of refractive index and molar refraction of {[(TeO ₂) _{0.7} (B ₂ O ₃) _{0.3}] _{0.7} [ZnO] _{0.3} } _{0.99} {Tm ₂ O ₃ } _{0.01} glass system before and after radiation process	5.97
5.100	Variation of molar and oxide ion polarizability of	5.97

$\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3\}_{0.01}$ glass system before and after radiation process

- | | | |
|-------|--|-------|
| 5.101 | Variation of refractive index and molar refraction of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{CeO}_2\}_{0.01}$ glass system before and after radiation process | 5.98 |
| 5.102 | Variation of molar and oxide ion polarizability of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{CeO}_2\}_{0.01}$ glass system before and after radiation process | 5.98 |
| 5.103 | Variation of optical basicity of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_{0.01}$ glass system before and after radiation process | 5.100 |
| 5.104 | Variation of metallization criterion of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{0.99}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_{0.01}$ glass system before and after radiation process | 5.101 |

LIST OF ABBREVIATIONS

TeO_2	Tellurium oxide
B_2O_3	Boron oxide
ZnO	Zinc oxide
Tm_2O_3	Thulium oxide
CeO_2	Cerium oxide
BOs	Bridging oxygens
NBOs	Non-bridging oxygens
Tm	Thulium
Ce	Cerium
ρ	Density
V_m	Molar volume
V_L	Longitudinal velocity
V_S	Shear velocity
L	Longitudinal modulus
G	Shear modulus
K	Bulk modulus
Y	Young's modulus
σ	Poisson's ratio
H	Microhardness
θ	Debye temperature
Z	Acoustic impedance
T_s	Softening temperature
M	Metallization criterion
E_{opt}	Optical band gap
n	Refractive index
R_m	Molar refraction
α_m	Molar polarizability
$\alpha_{\text{O}^{2-}}$	Oxide ion polarizability
Λ	Optical basicity
γ	gamma

CHAPTER 1

INTRODUCTION

This chapter deals with the research background and consists of a brief introduction to glass, tellurite glass, and all chemicals that were used in this research. Besides that, other important components of the study were also included, such as the problem statement, objectives of study, significance of study and hypothesis as well as scope and limitations.

1.1 Research background

1.1.1 Definition of glass

The word glass is derived from the Latin term *glæsum*, referring to a lustrous and transparent material. During early civilizations, the most significant properties attributed to glass are luster or shine, and in particular its durability when being exposed to natural elements (Varshneya, 2013). It is the oldest man-made material and its invention has enabled man to have a broad daylight in his protected environment (Aben and Claude, 2012).

According to the American Society for Testing Materials (ASTM) in 1945, glass can be defined as “an inorganic product of fusion which was cooled to a rigid condition without crystallizing”. However, this definition does not give a full explanation about glass, since several other ways to produce glass were discovered later. These include vapour deposition, sol-gel processing of solutions and neutron irradiation of crystalline materials. Both sol-gel processing and chemical vapour deposition are techniques that would avoid the normal high temperatures applied for glass formation.

Among the common characteristics of glass are, it does not have a long range, periodic atomic arrangement and it shows time-dependent glass transformation behaviour that makes it different when compared to the other types of solid. The absence of long-range order is the property that made glass known as an amorphous solid. From the above statement, glass can be fully defined as an amorphous solid that is completely lacking in long range, periodic atomic structure, and exhibiting a region of glass transformation behaviour (Shelby, 2005).

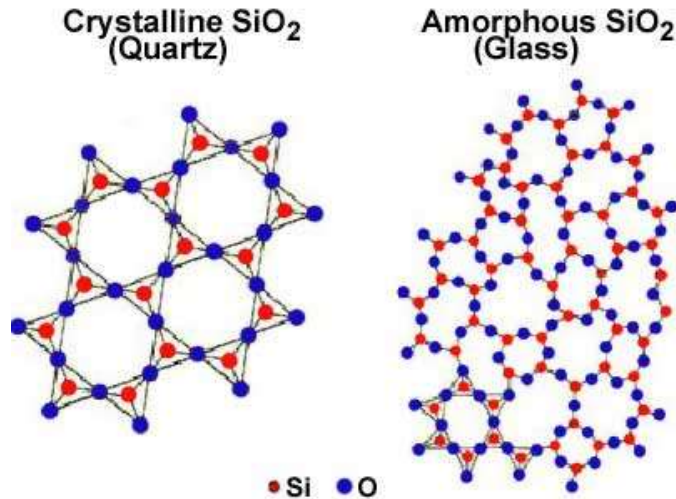


Figure 1.1: Network structure of crystalline and amorphous solid (Mayhugh, 2010)

1.1.2 Tellurite glass (TeO₂)

Tellurium dioxide is a conditional glass former which can form a glass on its own. The term ‘conditional’ is used as the process to produce the glass is very difficult if the chemical is not added with other chemical compounds. Before 1985, the studies on this type of glass are very scarce and unattractive to international attention (Rivera and Manzani, 2017). However, this situation recently has started to change when the TeO₂ glass was found to possess many unique properties as it was doped with transition metals or rare earth ions. Some advantages of tellurite glass are low melting points, high refractive index, high dielectric constant and good infrared transmission (Halimah et al., 2005a). Besides that, Podmaniczky (1976) and Warner et al., (1972) mentioned that it has low acoustic losses and slow-shear wave propagation velocity. Hence, they suggested that tellurite glass has a potential to be used in laser light modulators.

In terms of rare-earth doped tellurite glass, many researchers suggested that it has a potential to be used in optoelectronic areas. El-Mallawany et al., (2004) stated in their study that Er³⁺ doped tellurite-titanium-tungsten glasses are suitable to be applied as fibre amplifier since a broad emission spectrum can be detected at 1550 nm with a width of 121 nm and decay time 4.5 ms. In addition, the increment of overall upconversion fluorescence and relative increase in the intensity of red emission with respect to green emission make the glass a promising candidate for infrared amplifiers as well as for red and green upconversion emissions. Besides that, Er³⁺ - Yb³⁺ codoped TeO₂-PbF₂ oxyhalide tellurite glasses that was studied by Yang et al., (2014) was found to enhance the performance of amorphous silicon solar cell as they were coupled together.

1.1.3 Borate (B_2O_3)

Borate is also one of the glass formers which can form glass on its own. According to Kotz, (2016), borates are a large number of boron-containing oxyanions that are $(BO_3)^{3-}$ units. These units exist in trigonal planar BO_3 and tetrahedral BO_4 structural units which are connected in linear or cyclic arrangement. Borate can also be studied by obtaining information regarding two elements that are present in it namely boron and oxygen. In the periodic table, boron is the fifth element and categorized as a metalloid. Metalloid properties lie between metals and non-metals. It does not conduct electricity and heat as good as a metal like copper (Cu) but conducts better than non-metal such as sulphur (S) (Adair, 2007). Boron and oxygen in borates form strong covalent bonds and thus make them suitable to be used as a glass former.

Borate glass is known to have high transparency, low melting point, good rare-earth ion solubility and high phonon energy (Maheshvaran et al., 2013). Besides that, several researchers also stated that it has high chemical durability, thermal stability and cost effective properties (Reddy et al., 2015; Nurbaisyatul et al., 2014). These advantages make the glass suitable for various applications such as optical materials, low temperature sealing glasses and electronic devices (Chimalawong et al., 2010; Rejisha et al., 2016; Beckmann et al., 2013). However, borate glass is also known to possess a hygroscopic nature where it can react with atmospheric water. This, in turn, limits the glass applications. In order to overcome this problem, other chemical oxides are added into the glass system such as alkali metal, heavy metal and rare-earth oxides.

1.1.4 Zinc oxide (ZnO)

Zinc oxide is an inorganic compound which is white in colour (powdered form) and insoluble in water. It is widely used as additive in materials such as plastic, glass, lubricants and cement. It is naturally found as mineral zincite which is yellow to red in colour and usually contains a certain amount of manganese (Klingshirn, 2007). Besides that, ZnO can also be synthetically produced via direct, indirect and wet chemical processes (Porter, 1991). In the periodic table, zinc is the first element in group 12 and classified as a transition metal. Some general properties of transition metals are high melting points, good electrical conductivity and have a moderate-to-extreme hardness (Petrucci et al., 2010). In addition, Helmenstine, (2016) stated that transition metal also has low ionization energy, positive oxidation states and exhibits metallic luster. Transition metal ions are used as dopants in glasses mainly due to two reasons; 1) they have defined and sharp energy levels that can serve as structural probes for the dopant environment, and 2) modifications of energy level structure due to dopants insertion may lead to interesting applications, such as new lasers and luminescence materials (Al-Shamiri and Eid, 2012).

Many previous researchers have reported the improvement in the properties of a material when zinc oxide (ZnO) is added into it. Kundu et al., (2014) mentioned that the insertion of ZnO into glass system produces stability and increases the glass forming ability. Furthermore, Azlan et al., (2013) stated that ZnO can reduce the

melting point and optical band gap as well as increase the refractive index of glass. On top of that, it can also act both as the network former and network modifier with a certain composition.

1.1.5 Thulium oxide (Tm₂O₃)

Thulium with chemical formula Tm was firstly discovered by Per Teodor Cleve, a Swedish chemist, who separated it from an impure sample of erbia in 1879 (Gagnon, n.d.). It is a pale green colour compound and can also be prepared synthetically by burning thulium metal in air or decomposition of their oxyacid salts such as thulium nitrate. Tm is categorized as a rare-earth element which is found in very small amount in the vast majority of the geological environment (Atwood, 2012). In lanthanide series, thulium is the thirteenth element which is situated between erbium (Er) and ytterbium (Yb). The common oxidation state for Tm is +3 while it is also available in +2. According to Bonnelle and Spector, (2015), thulium is found in the trivalent state in TmAl₂, TmS, and Tm₂O₃ while it can exist in both trivalent and divalent states in TmTe and TmSe. A few known uses of Tm and its isotopes are as a radiation source in portable X-ray equipment and dopant in ceramic magnetic materials used in microwave equipment. (Weast and Lide, 1989).

In terms of thulium oxide specifically, it can be used in numerous applications such as X-ray devices, phosphors, atomic reactors and semiconductors (Sidorowicz et al., 2016). Meanwhile, Cho et al., (2000) stated that the insertion of Tm³⁺ into tellurite glass network produces an excellent candidate material for efficient optical fibre amplifiers at 1.47 μm region. Besides that, Tm-doped materials also have drawn much interest due to its ability to produce blue coherent radiation pumped by infrared lasers (Santos et al., 2001). A blue laser is very useful as it can be applied in many fields such as submarine communications and optical data storage (Hanna et al., 1990). Furthermore, lasers containing thulium are also used in surgery, dentistry, atmospheric testing and remote sensing.

1.1.6 Cerium oxide (CeO₂)

Cerium (Ce) is a soft, ductile and malleable metal which is very reactive in air or water ("Cerium," n.d.). It is the second element in lanthanide series and located between lanthanum (La) and Praseodymium (Pr). Among the rare earth elements, Ce is the most abundant and making up 0.0046% of the Earth crust. It was firstly discovered in 1803 by Klaproth, Berzalius and Hisinger. A number of minerals found to contain cerium include allanite (also known as orthite), samarskite, monazite, cerite and bastnasite. Besides that, it can also be produced synthetically by metallothermic reduction techniques such as reducing cerous fluoride with calcium or using electrolysis of molten cerous chloride. These techniques can produce high-purity cerium ("Cerium," 2016). This element is extensively used in the manufacture of pyrophoric alloys for cigarette lighters. Furthermore, it is used in carbon-arc lighting, especially in the motion picture industry and also useful as a catalyst in petroleum refining, metallurgical and nuclear applications.

Nowadays, cerium oxide also called ceria is the most common form of cerium that is widely used in many applications. The main use of CeO_2 is as a polishing compound for glass, replacing rouge as it can polish much faster. Moreover, it is also an important constituent of incandescent gas mantles. The other emerging application is as a hydrocarbon catalyst in the self-cleaning oven where its incorporation into the oven walls can prevent cooking residues collection. In terms of cerium-doped glasses, Singh and Singh, (2011) reported that they are used in various applications such as laser active media, radiation protection of fibre optic materials and scintillation materials.

1.2 Problem statement

Nowadays, numerous studies have been done on the properties of glass and its potential applications. Based on the previous researches, it is widely known that the characteristic of glass can be controlled via the addition of chemical oxides. This is due to the fact that each chemical oxide possesses unique properties which can then alter the glass overall characteristics. The insertion of zinc oxide (ZnO) for example, can decrease tellurite glass density (Kaur et al., 2010; Redman and Chen, 1967) while the incorporation of aluminium oxide (Al_2O_3) causes the elastic moduli of tellurite-based glass to increase (Balaji et al., 2014). Besides that, it is also important to note that this modification is not only dependent on the modifier oxide but also on the overall composition of the glass system.

The doping of glass with rare earth oxides grabs the researchers' attention due to their ability to enhance the optical properties of glass. This is attributed to the existence of 4f electrons in these elements. According to Reddy et al., (2015), the effect of ligand environment on the 4f shell is minimum as rare earth is embedded into a solid matrix. This is caused by 4s and 5p shells which effectively shield the 4f shell. As a result, it is easier for the 4f electrons to be ejected from the atom and produce the optical transitions that do not exist in the other compound. However, some problems arise when the rare earth oxides are added to a high composition, where the glass samples tend to become more fragile and in some cases attain a crystalline nature. Thus, another method was used in the present study to alter the glass properties which is an exposure to gamma radiation. Using this method, it is believed that the glass characteristic can be further modified while at the same time maintain a low percentage of rare earth ions.

Several studies were conducted on borotellurite-based glass by previous researchers. Maheshvaran et al., (2013) mentioned that B_2O_3 - TeO_2 glass with the addition of alkali oxide produce glasses with low phonon energy, high refractive index and high optical non-linearity. Besides that, it also stated that borotellurite-based glass is suitable to be used in the fabrication of various new optical devices. Recently, there have been several studies that investigated on zinc borotellurite glass (Eevon et al., 2016; Faznny et al., 2016; Hazlin et al., 2017), but different types of rare earth oxides are used in their study. There is still lack of research that presented on thulium and cerium-doped zinc borotellurite glass system. Moreover, exposing thulium and cerium-doped zinc borotellurite glass to gamma radiation also has not been done by

other researchers before. Hence, this study was conducted in order to study the structural, elastic and optical properties of the glass system. Furthermore, the influences of gamma radiation to the properties of the prepared glasses were also studied. All the data are presented in this thesis.

1.3 Objectives of study

This research was conducted based on four clear and concise objectives. The objectives are stated below;

1. To study the effect of Tm_2O_3/CeO_2 on elastic properties of zinc borotellurite glass system.
2. To analyze the effect of Tm_2O_3/CeO_2 on optical properties of zinc borotellurite glass system.
3. To determine the elastic properties of Tm_2O_3/CeO_2 doped zinc borotellurite glass using theoretical approach and compare with the experimental values.
4. To investigate the effect of gamma radiation on elastic and optical properties of Tm_2O_3/CeO_2 doped zinc borotellurite glass.

1.4 Hypotheses

Based on the above objectives, the hypotheses for this study are;

1. The addition of Tm_2O_3 and CeO_2 into glass samples are expected to enhance the elastic properties of the glass by increasing the elastic moduli, microhardness and Debye temperature while decreasing the value of Poisson's ratio. This is because rare earth ion causes the formation of bridging oxygen that will increase the rigidity of the glass samples.
2. These rare earth additions are expected to improve the optical properties of the glass by decreasing the energy band gap while increasing molar polarizability, molar refraction and also refractive index. The advantage of rare earth ion that has 4f electron is the contributing factor to the enhancement of these properties.
3. It is expected that the experimental and theoretical data of elastic properties are in a close range.
4. The exposure to gamma radiation is expected to change the structure, elastic and optical properties of glass samples. Gamma radiation will break the network bond in the structure and lead to the changes in its properties.

1.5 Scope and limitations

The objectives of this study are achieved by following the scope of the study stated below;

1. The preparation of the glasses was done by using melt-quenching technique and based on the stoichiometric equation: $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}(\text{ZnO})_{0.3}\}_{1-x}\{\text{Tm}_2\text{O}_3/\text{CeO}_2\}_x$. Tm_2O_3 and CeO_2 act as the dopants and vary from 0 to 0.05 mol.
2. The structures of glasses were analyzed using the Fourier Transform Infrared Spectroscopy (FTIR) to study the chemical bonding of the glasses and X-ray Diffraction (XRD) to confirm their amorphous nature.
3. The ultrasonic technique was used to obtain the ultrasonic wave velocity of the glass. Then, the longitudinal and shear velocities were used to determine the elastic properties of the glasses.
4. The optical properties of the glasses were investigated by using UV-VIS Spectrophotometer. Then, the optical parameters were calculated, such as optical band gap, Urbach energy, refractive index, molar and oxide ion polarizability and metallization criterion.
5. The effects of gamma radiation on the glasses were studied by exposing glasses containing 0.01 mol of dopant concentration to Co-60 gamma source with radiation doses ranging from 10 until 35 kGy.

1.6 Significance of study

Nowadays, glass is needed not only for windows and doors but also used as photonic, optical and electronic devices. Some studies about rare earth doped glass were found to enhance the properties of glass, especially in terms of its optical properties. Since then, there was a growing interest among researchers to study about rare earth doped-glass in order to produce glass with better properties.

Gamma radiation was found to be one of the factors that can modify the optical and elastic properties of glass. Thus, the outcome of this study may provide a deeper knowledge about the properties of the new composition of glasses and influence of gamma radiation to them. This research can be a guiding reference for further research on elastic and optical properties of rare earth-doped glass in the educational field and also in industrial applications.

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