



**UNIVERSITI PUTRA MALAYSIA**

**PHYSICAL CHARACTERIZATION OF LEAD BISMUTH BORATE AND  
LEAD BISMUTH PHOSPHATE GLASSES**

**HAMEZAN BIN MUHAMMAD @ AHMAD.**

**FS 2005 13**

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**By**

**HAMEZAN BIN MUHAMMAD @ AHMAD**

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

**December 2005**



*In The Name of Allah, The Beneficent, The Merciful*

*This Thesis is Dedicated to My Beloved Dad, Mom and Family*



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

**PHYSICAL CHARACTERIZATION OF LEAD BISMUTH BORATE AND  
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**HAMEZAN BIN MUHAMMAD @ AHMAD**

**December 2005**

**Chairman: Associate Professor Sidek Haji Abdul Aziz, PhD**

**Faculty: Science**

Systematic series of lead bismuth borate ( $\text{PbO} - \text{Bi}_2\text{O}_3 - \text{B}_2\text{O}_3$ ) and lead bismuth phosphate ( $\text{PbO} - \text{Bi}_2\text{O}_3 - \text{P}_2\text{O}_5$ ) glasses were prepared using melt quenching technique, where  $\text{PbO}$ ,  $\text{Bi}_2\text{O}_3$ ,  $\text{B}_2\text{O}_3$  and  $\text{P}_2\text{O}_5$  contents changed in every series based on their weight percentage. Some physical properties were measured and their amorphous natures were confirmed earlier by the X-ray diffraction technique.

The experimental results showed that the density ( $\rho$ ) of both glasses increased, for examples from  $3920 \text{ kg/m}^3$  to  $6325 \text{ kg/m}^3$  for A1 – A5 series in  $\text{PbO} - \text{Bi}_2\text{O}_3 - \text{B}_2\text{O}_3$  glasses and from  $4331 \text{ kg/m}^3$  to  $5698 \text{ kg/m}^3$  for E1 – E5 series in  $\text{PbO} - \text{Bi}_2\text{O}_3 - \text{P}_2\text{O}_5$  glasses. This was due to the replacement of  $\text{Bi}_2\text{O}_3$  and  $\text{PbO}$  in the  $\text{B}_2\text{O}_3$  and  $\text{P}_2\text{O}_5$  in glassy networks. Additional increment of  $\text{Bi}_2\text{O}_3$  and  $\text{PbO}$  in both types of glasses causing more discontinuity and hence, decreased in their rigidity and velocity. Meanwhile,



there was also a similar pattern in elastic moduli in both glass systems, where the values increased at the earlier stage and then decreased subsequently. Both Young's and bulk modulus were related to the cross-linking density with large influence on the propagation of ultrasonic velocities. All glass samples were also found to have crosslink density of 1 and Poisson's ratio  $\sim 3$  which was typical for the  $B_2O_3$  and  $P_2O_5$  glasses.

In optical properties for both types of glasses, it was found that the shifting of wavelength was related to the amount of production of the non-bridging oxygen (NBO). The existence of less disorder in phosphate network contributed to higher values of glass optical band gap ( $E_{opt}$ ). Conversely, the introduction of PbO and  $Bi_2O_3$  cause great disorder happen in the borate network which results in lower  $E_{opt}$  values. In this study, the values of  $E_{opt}$  decreased uniformly with increasing content of PbO and  $Bi_2O_3$  for examples from 2.61 eV to 2.25 eV for B1 – B5 series in PbO –  $Bi_2O_3$  –  $B_2O_3$  glasses and from 3.71 eV to 3.06 eV for G1 – G4 series in PbO –  $Bi_2O_3$  –  $P_2O_5$  glasses. The increases in NBOs will be accompanied by an increase in polarizability and refractive index ( $n$ ). In most cases, the variation of  $n$  increases when the molar volume ( $V_m$ ) decreases, however for PbO –  $Bi_2O_3$  –  $B_2O_3$  glasses, the increasing value of  $n$  for an example from 1.62 to 1.86 for C1 – C5 series is accompanied by an increased in  $V_m$ . This discrepancy can be explained by assuming the increase in both of the  $V_m$  and  $\rho$ , was attributed to change occurred in the volume concentration of  $BO_3$  units.

Results from thermal studies of the glass showed that values for glass transition temperature ( $T_g$ ) was closely related to the chemical bond in the



system. For  $\text{PbO} - \text{Bi}_2\text{O}_3 - \text{B}_2\text{O}_3$  glasses, the ionic bond character became more dominant in the system with the addition of more  $\text{Pb}^{2+}$  and  $\text{Bi}^{3+}$  and hence decreases the  $T_g$  of sample. However, in  $\text{PbO} - \text{Bi}_2\text{O}_3 - \text{P}_2\text{O}_5$  glasses, the addition of  $\text{Pb}^{2+}$  and  $\text{Bi}^{3+}$  not only failed to weaken the covalent character in P–O–P bonds, but strengthened it further which leads to an increment in  $T_g$  values for an example from  $309^\circ\text{C}$  to  $352^\circ\text{C}$  for F1 – F4 series.

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

**PENCIRIAN SIFAT FIZIKAL BAGI KACA PLUMBUM BISMUTH BORAT  
DAN PLUMBUM BISMUTH FOSFAT**

Oleh

**HAMEZAN BIN MUHAMMAD @ AHMAD**

**Disember 2005**

**Pengerusi: Profesor Madya Sidek Haji Abdul Aziz, PhD**

**Fakulti: Sains**

Siri sistematik kaca plumbum bismuth borat ( $\text{PbO} - \text{Bi}_2\text{O}_3 - \text{B}_2\text{O}_3$ ) dan plumbum bismuth fosfat ( $\text{PbO} - \text{Bi}_2\text{O}_3 - \text{P}_2\text{O}_5$ ) telah disediakan melalui teknik pelindapan leburan, di mana kandungan  $\text{PbO}$ ,  $\text{Bi}_2\text{O}_3$ ,  $\text{B}_2\text{O}_3$  dan  $\text{P}_2\text{O}_5$  telah berubah dalam setiap siri berdasarkan kepada peratusan berat bahan. Beberapa ciri fizikal telah diukur dan sifat amorfus bahan terlebih dahulu telah disahkan menggunakan teknik pembelauan sinar-X.

Keputusan ujikaji menunjukkan ketumpatan ( $\rho$ ) bagi kedua jenis kaca telah meningkat, sebagai contoh dari  $3920 \text{ kg/m}^3$  ke  $6325 \text{ kg/m}^3$  untuk siri A1 – A5 dalam kaca  $\text{PbO} - \text{Bi}_2\text{O}_3 - \text{B}_2\text{O}_3$  dan dari  $4331 \text{ kg/m}^3$  ke  $5698 \text{ kg/m}^3$  untuk siri E1 – E5 dalam kaca  $\text{PbO} - \text{Bi}_2\text{O}_3 - \text{P}_2\text{O}_5$ . Ini berlaku hasil penggantian  $\text{Bi}_2\text{O}_3$  dan  $\text{PbO}$  ke dalam rangkaian kaca  $\text{B}_2\text{O}_3$  dan  $\text{P}_2\text{O}_5$ . Pertambahan  $\text{Bi}_2\text{O}_3$  dan  $\text{PbO}$  di dalam kedua-dua jenis kaca, menyebabkan banyak ketidaksinambungan dan dengan itu, telah berlaku penurunan dalam sifat



kekakuan dan halaju. Sementara itu, terdapat juga corak sama dalam modulus elastik bagi kedua-dua sistem kaca, di mana nilai-nilai telah meningkat pada peringkat awal dan kemudiannya menurun. Modulus Young dan pukal adalah berkait kepada ketumpatan pemautilangan dengan memberi kesan besar ke atas penyebaran halaju ultrasonik. Kesemua sampel kaca mempunyai ketumpatan pemautilang bersamaan 1 dan nisbah Poisson  $\sim 3$  di mana ini adalah tipikal untuk kaca  $B_2O_3$  dan  $P_2O_5$ .

Di dalam pencirian optik untuk kedua-dua jenis kaca, didapati bahawa anjakan jarak gelombang adalah berkait dengan jumlah penghasilan oksigen tanpa titian (NBO). Kewujudan kurang ketidakseragaman dalam rangkaian fosfat telah menyumbang kepada nilai-nilai sela jalur optik ( $E_{opt}$ ) yang tinggi. Sebaliknya, pengenalan PbO dan  $Bi_2O_3$  menyebabkan lebih ketidakseragaman berlaku di dalam rangkaian borat yang mana menghasilkan nilai-nilai  $E_{opt}$  yang rendah. Melalui kajian ini, nilai-nilai  $E_{opt}$  telah menurun secara seragam dengan peningkatan kandungan PbO dan  $Bi_2O_3$  sebagai contoh dari 2.61 eV ke 2.25 eV untuk siri B1 – B5 dalam kaca PbO –  $Bi_2O_3$  –  $B_2O_3$  dan dari 3.71 eV ke 3.06 eV untuk siri G1 – G4 dalam kaca PbO –  $Bi_2O_3$  –  $P_2O_5$ . Peningkatan dalam NBO disertai dengan kenaikan dalam kebolehkutuban dan indeks biasan ( $n$ ). Di dalam kebanyakan situasi, variasi  $n$  meningkat apabila isipadu molar ( $V_m$ ) menurun, bagaimanapun bagi kaca PbO –  $Bi_2O_3$  –  $B_2O_3$ , peningkatan nilai  $n$  sebagai contoh dari 1.62 ke 1.86 untuk siri C1 – C5 telah disertai dengan peningkatan dalam  $V_m$ . Ketidapatuhan ini dapat dijelaskan dengan menganggap peningkatan dalam kedua-dua  $\rho$  dan  $V_m$ , adalah merujuk kepada perubahan yang telah berlaku di dalam kepekatan isipadu unit-unit  $BO_3$ .





Keputusan dari kajian ciri terma untuk kaca telah menunjukkan bahawa nilai-nilai suhu transisi kaca ( $T_g$ ) adalah berkait rapat dengan ikatan kimia di dalam sistem. Bagi kaca  $\text{PbO} - \text{Bi}_2\text{O}_3 - \text{B}_2\text{O}_3$ , sifat ikatan ionik telah menjadi lebih dominan di dalam sistem dengan penambahan lebih banyak  $\text{Pb}^{2+}$  dan  $\text{Bi}^{3+}$  dan menyebabkan penurunan pada  $T_g$  sampel. Bagaimanapun, dalam kaca  $\text{PbO} - \text{Bi}_2\text{O}_3 - \text{P}_2\text{O}_5$ , penambahan  $\text{Pb}^{2+}$  dan  $\text{Bi}^{3+}$  bukan sahaja telah gagal melemahkan sifat kovalen pada ikatan  $\text{P-O-P}$ , malah telah menjadikannya lebih kuat di mana membawa kepada peningkatan dalam nilai-nilai  $T_g$ , sebagai contoh dari  $309^\circ\text{C}$  ke  $352^\circ\text{C}$  untuk siri F1 – F4.



## ACKNOWLEDGEMENTS

I wish to thank the many people who have, in one way or the other, made this thesis possible. First of all, my project supervisor Associate Professor Dr. Sidek Hj. Abdul Aziz, for all his help, support and valuable discussions, and also from whom I have gained many new insight.

To all my co-supervisor Associate Professor Dr. Zaidan Abdul Wahab and Professor Dr. Kaida Khalid, all at the Department of Physics, Faculty of Science, Universiti Putra Malaysia (UPM), for all their support and guidance during my research study. My special thanks to Associate Professor Dr. Zainal Abidin Talib for reading part of this thesis and for his valuable comments concerning on optical analysis.

I also wish to thank all members of the Ultrasonic Research Laboratory, Sheik Azrif, Liu Wei Wen, Faizal, Azhar, Che Wan, my seniors Mr. Khamirul, Mdm. Halimah, Mdm. Rosmawati, and to all my friends at Department of Physics, especially Emma, Loh, Iskandar, Asri, Rudy and Walter for help, discussions and for being the best of friends. I am especially grateful to Ms. Ezarul Faradianna for helping me during my thesis writing and pointing out many errors.

I am also grateful to the MOSTE for their financial support in this research under the IRPA grant vote: 09-02-04-0550-EA001. Also, I would like to thank all the staff at Department of Physics for managing and ordering materials used in this research.



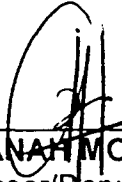
I certify that an Examination Committee met on 5<sup>th</sup> December 2005 to conduct the final examination of Hamezan Bin Muhammad @ Ahmad on his Master of Science thesis entitled "Physical Characterization of Lead Bismuth Borate and Lead Bismuth Phosphate Glasses" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulation 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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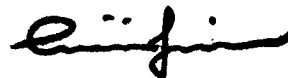


This thesis submitted to the Senate of Universiti Putra Malaysia has been accepted as fulfillment of the requirements for the degree of Master of Science. The members of the Supervisory Committee are as follows:

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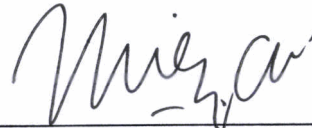
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## DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.



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**HAMEZAN BIN MUHAMMAD @ AHMAD**

Date: **28 DEC 2005**

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## LIST OF ABBREVIATIONS/NOTATIONS/GLOSSARY OF TERMS

DTA	Differential Thermal Analysis
DSC	Differential Scanning Calorimeter
FTIR	Fourier Transform Infrared Spectroscopy
IR	Infrared
NBO	Non-bridging oxygen
NMR	Nuclear Magnetic Resonance
TMA	Thermo Mechanical Analyzer
UV	Ultraviolet
Vis	Visible
XAFS	X-ray Absorption Fine Structure
XPS	X-ray Photoelectron Spectroscopy
$A$	Absorbance
$B$	Bulk modulus
$C_p$	Heat capacity
$C_{11}$	Longitudinal modulus
$C_{44}$	Shear modulus
$E$	Electric field
$E_{opt}$	Optical band gap
$\Delta E$	Urbach energy
$F$	Applied force
$J$	Current density
$L$	Length
$M$	Molecular weight



$M_s$	Mean velocity
$N_A$	Avogadro's number
$P$	Power of light
$T$	Transmittance
$T_c$	Glass crystallization temperature
$T_g$	Glass transition temperature
$T_m$	Glass melting point
$T_f$	Fictive temperature
$V$	Velocity
$V_L$	Longitudinal velocity
$V_s$	Shear velocity
$V_m$	Molar volume
$W_{air}$	Weight of sample in the air
$W_{acetone}$	Weight of sample in acetone
$Y$	Young modulus
$Z$	Number of atoms
$Z$	Acoustic impedance
$c$	Speed of light in vacuum
$d$	Thickness of the sample
$f$	Frequency
$h$	Planck's constant
$k$	Boltzmann's constant
$n$	Index of refraction
$q$	Cooling rate
$v$	Speed of light in a medium





$\alpha$	Absorption coefficient
$\alpha_T$	Thermal expansion
$\delta_{ij}$	Kronecker delta
$\varepsilon$	Linear strain
$\theta$	Angle of refraction
$\theta_D$	Debye temperature
$\lambda$	Wavelength
$\rho$	Mass density
$\rho_{\text{acetone}}$	Absolute density of acetone
$\rho_{\text{sample}}$	Density of sample
$\sigma$	Poisson's ratio
$\sigma$	Conductivity
$\sigma$	Linear stress
$\omega$	Angular frequency
$\hbar$	Reduced Planck's constant
$\kappa_T$	Compressibility

