PHYSICAL AND DIELECTRIC PROPERTIES OF BORO-TELLURITE GLASS DOPED WITH COBALT AND IRON OXIDES

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By

ERNY SAFARINA SAAD

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

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DEDICATION

Specially dedicated to:

My Husband,
My Son,
My Father,
My Mother,
My Sisters,
My Brothers,
My Lecturers,
My Friends,

for their encouragement and support. ☺

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

PHYSICAL AND DIELECTRIC PROPERTIES OF BORO-TELLURITE GLASS DOPED WITH COBALT AND IRON OXIDES.

By

ERNY SAFARINA BINTI SAAD

January 2012

Chair: Halimah Mohamed Kamari, PhD

Faculty: Science

The glass system with composition $[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{1-x}[Co_3O_4]_x$ with x= 0.01, 0.02, 0.03, 0.04, 0.05 and 0.06, and $\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.95}[Co_3O_4]_{0.05}\}_{1-y}\{Fe_2O_3\}_y$ with y= 0.01, 0.02, 0.03, 0.04, 0.05 and 0.06 has been successfully prepared by the melt quenching technique. The amorphous nature of glass system was determined by using XRD measurement. The glass transition temperature, (Tg), was determined using the DTA technique at room temperature. Density of variation glasses sample was measured at room temperature using the Archimedes technique. The values of molar volume were calculated from density data. The infrared absorption spectra of the glasses sample in the wavenumber range of 280-4000 cm^{-1} were recorded by using Fourier Transform Infrared Spectroscopy. The magnetic behaviour in the glass system was studied using VSM (vibrating sample magnetometer) at room temperature with magnetic field range ±15kOe. The susceptibility was determined from the relationship between magnetic field, H (Oe) and magnetic moment/mass, M (emu/mass). The magnetization, of the glass sample increases with increasing of magnetic field, H.

Dielectric properties have been investigated in the frequency range 10^{-2} to 10^{3} Hz and temperature range 30 °C-200 °C. From this measurement, the dielectric properties such as dielectric permittivity, ε', and dielectric loss, ε", was determined. The ε' and ε" of glasses sample increases with temperature and decreases with frequency. The activation energy of dielectric properties was determined from normalization curve using the Arrhenius equation. The activation energy of both glasses sample increases with increasing cobalt and iron oxide content. The conductivity, σ of glasses sample also been calculated from equation, $\sigma_{ac} = \omega\varepsilon_0\varepsilon''$. The value of dc conductivity, σ_{dc} was determined from "ac universal power law", and the value activation energy of σ_{dc}, has been calculated using the Arrhenius implied. The activation energy of σ_{dc} was increased with increasing of cobalt and iron oxide. The activation energy of dc conductivity was almost equal with dielectric loss relaxation indicating the same species took part in both the process. The dielectric behaviour of these glass systems also has been analyzed using electric modulus, M and impedance, Z, formalism. The graph of M" and Z" peaks were found overlapping each other. This behaviour indicated a long range conductivity process appears in this glasses sample. For first series glasses sample, the value of activation energy of impedance plot, Z" vs. Z', increases with increasing of cobalt oxide content. For the second series glass samples, the curve of Z" vs. Z' plot will compose of two semicircle with increases of iron oxide content. The activation energy for second series glass samples decreases with increasing iron oxide content.

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

CIRI FIZIKAL DAN DIELEKTRIK BAGI KACA BORO-TELLURITE APABILA DITAMBAH DENGAN KOBALT DAN FERUM OKSIDA.

Oleh

ERNY SAFARINA BINTI SAAD

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Sistem kaca dengan pelbagai komposisi bagi $[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{1-x}[Co_3O_4]_x$ dengan x= 0.01, 0.02, 0.03, 0.04, 0.05 dan 0.06, dan $\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.95}[Co_3O_4]_{0.05}\}_{1-y}\{Fe_2O_3\}y$ dengan y= 0.01, 0.02, 0.03, 0.04, 0.05 dan 0.06 dengan jayanya disediakan menggunakan kaedah peleburan lazim dan sepuh lindap. Sifat bahan amorfus bagi kaca ditentukan dengan menggunakan pengukuran XRD. Suhu transisi kaca, Tg ditentukan dengan menggunakan teknik Analisis Pembebezaan Termal, DTA, pada suhu bilik. Ketumpatan bagi pelbagai variasi sampel kaca,diukur pada suhu bilik menggunakan teknik Archimedes. Nilai isipadu molar telah dikira daripada nilai ketumpatan kaca yang diperolehi. Spektra penyerapan inframerah bagi sampel kaca dalam julat 280-4000cm^{-1} telah direkodkan menggunakan alatan spektroskopi infra merah, FTIR. Sifat- sifat magnet dalam sampel kaca telah dikaji menggunakan sistem pengukuran VSM *(vibrating sample magnetometer)* pada suhu bilik dengan julat medan magnet ±15kOe. Kerentanan magnetik telah ditentukan daripada hubungan antara medan magnet, H(Oe)

dan momen magnet/jisim, M(emu/mass). Kemagnetan kaca meningkat dengan kadar peningkatan medan magnet. Sifat dielektrik telah diselidik dalam julat frekuensi dari 10^{-2} hingga 10^{3} Hz pada julat suhu 30 °C-200 °C. Daripada pengukuran ini, perilaku sifat dielektrik seperti dielektrik permitiviti, ε' dan faktor kehilangan dielektrik ,ε" telah didapati. ε' dan ε" didapati meningkat dengan peningkatan suhu dan menurun dengan peningkatan frekuansi. Tenaga pengaktifan bagi sifat dielektrik ditentukan dari lengkungan normalisasi menggunakan persamaan Arrhenius. Tenaga pengaktifan bagi kedua-dua sampel kaca meningkat dengan kadar peningkatan kobalt dan ferum oksida. Kekonduksian, σ bagi sampel kaca juga telah dikira daripda persamaa, $\sigma_{ac} = \omega\varepsilon_0\varepsilon''$. Nilai bagi arus terus, σ_{dc}, telah ditentukan daripada "ac power law", dan nilai tenaga pengaktifan, σ_{dc} dikira menggunakn persamaan Arhenius. Tenaga pengaktifan kekonduksian arus terus, σ_{dc} , meningkat dengan peningkatan kandungan kobalt dan ferum oksida. Nilai tenaga pengaktifan kekonduksian, σ_{dc} adalah hampir sama dengan tenaga pengaktifan bagi kehilangan dielektrik menunjukkan spesies yang sama mengambil bahagian dalam kedua-dua process. Sifat dielektrik bagi kedua-dua sistem kaca ini juga dianalisa menggunakan modulus, M dan impedance spektroskopi, Z. Puncak graf M "dan Z" didapati bertindih antara satu sama lain. Ini menunjukkan kekonduksian proses jangka panjang berlaku dalam sampel ini. Bagi sampel kaca siri pertama, nilai tenaga pengaktifan bagi impedan plot, Z" melawan Z', meningkat dengan kadar peningkatan kobalt oksida. Bagi sampel kaca bagi siri kedua, lengkungan dari plot Z" terhadap Z 'akan menjadi dua semibulatan dengan peningkatan kandungan ferum oksida. Bagi nilai tenaga pengaktifan bagi sampel kaca siri kedua menurun dengan peningkatan kadar kandungan ferum oksida.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisor Committee were as follows:

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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

ERNY SAFARINA SAAD

Date: 10 January 2012

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LIST OF ABBREVIATIONS

β	Beta
Å	Angstrom
ρ	Density
λ	Wavelength
θ	Theta
FT-IR	Fourier Transform Infrared
χ	Susceptibility
B	Induction
H	Applied field
π	Pi
m	Magnetic moment
v	Volume of solid
μ_o	Permeability of free space
μ	Permeability
ε^*	Complex permeability
ε’	Real part of permittivity
ε”	Imaginary part of permittivity
TD	Time-domain
FD	Frequency-domain
ω	Angular frequency
χ'	Real part of susceptibility
χ''	Imaginary part of susceptibility

∞	Infinity
ε_0	Permittivity of free space($8.854 \times 10^{-12} Fm^{-1}$)
M*	Complex electrical modulus
M'	Real part electrical modulus
M''	Imaginary part electrical modulus
C*	Complex capacitor
i	Imaginary number (√ -1)
G	Conductance
Y*	Complex admittance
Z*	Complex impedance
ωp	Loss peak frequency
C	Capacitance
R	Resistance
α	Alpha
E	Electric field
ε_r	Relative permittivity
P	Polarization
Co	Capacitance of the air-filled parallel plate capacitor
σ	Conductivity of the medium
τ	Relaxation time

CHAPTER 1

INTRODUCTION

1.1 Introduction

Boro-tellurite glass is one of the amorphous materials. It preparation steps are involving the quenching of molten or liquid cooling of the melt at a certain rate and temperature. Due to intensive application in nowadays, it's being the interesting glasses to study.

In this chapter will introduce about the glass, important of Tellurite glass, introduction to the cobalt and iron oxide, scope of study, problem statement and the objectives of this study.

1.2 Introduction of the Glass

Many types of glasses will produce by melt quenching technique. The phenomenon due to the quenching are called the glass transition phenomenon. The glass transition phenomenon will occur only at certain temperature called the glass transition temperature (Rahim Sahar, 2000).

The others definition of glass is, glass are an inorganic product of fusion which has cooled to rigid condition without crystallizing. Thus, non-crystalline solids can be made by deposition from the vapor phase, or by sputtering in low pressure system, and these have the same chemical composition as, and apparently identical properties to, glass produced by cooling from the melt. The committee of the U.S. National Research Council recently proposed a more general definition there is; glass is an x-ray amorphous material which exhibits the glass transition, this being exhibits the glass transition, this being defined as that phenomenon in which a solid amorphous phase exhibits with changing temperature a more or less sudden change in the derivative thermodynamic properties, such as heat capacity and expansion coefficient from crystal-like to liquid-like values.

Since the glass is amorphous materials, in x-ray diffraction experiment does not reveal any sharp peaks but only broad humps the nature of the glass system (Rawson, 1980). The Figure 1.1 shows the different of atoms arrangement in amorphous and crystal state.

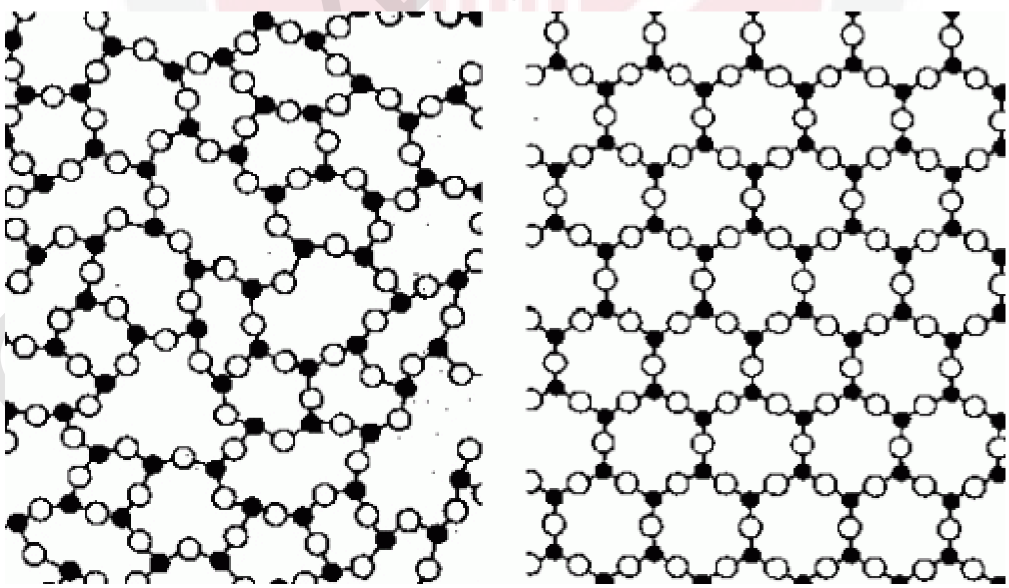

Figure 1.1: Amorphous structure of a glassy solid (left) and lattice structure of a crystalline solid (right)
(www.e-education.psu.edu/eart520/node/1689)

1.3 Important of Tellurite glasses

During two decade, tellurite glasses have been studied for many years due to numerous applications and great research effort. Therefore, tellurite glasses are now under consideration in many others applications. It has potential application in pressure sensors or as a new laser hosts because of their exhibited a range of unique properties (El-Mallawany, 2002). The tellurite glasses also interesting on account of their high values of refractive index and dielectric constant (Rawson, 1980). Munoz-Martin *et al.* (2011) state, tellurite glass is the promising materials for the development of integrated photonic devices due to their interesting in physical and chemical properties.

In important field of application like telecommunication technology, laser and optical fiber technology and optical device technology, tellurium glasses are be the ideal materials. This is due to their properties in high refractive index (in the range from 1.85 to 2.2), high non-linear third order optical susceptibility (50 times higher than one of SiO_2 systems), high transmittance from ultraviolet to near infrared, low transition temperature and electrical semi conductivity. It is also important for high ionic conductivity (Rada *et al.*, 2011).

With containing transition metal ions, it been observed that the TeO_2 have higher electronic conductivity than this glasses based on other conventional network formers such as P_2O_5, B_2O_3 and other glass former (Pan and Ghosh, 1999).

1.4 Introduction to Cobalt and Iron oxide

Transition metal glass such as iron ions, have a strong bearing on optical, magnetic and electrical properties. The environment of iron ions in various inorganic glass system such as silicate, borate, phosphate, germanate and tellurite, are available in a large number of interesting studies. In the glasses matrices, these ions exist in different valence states with different coordinations. An example, as Fe^{2+} with octahedral and as Fe^{3+} with both, tetrahedral and octahedral environment. Both of these ions are well-known paramagnetic ions. A large magnetic anisotropy of Fe^{2+} is due to its strong spin-orbit interaction of the 3d orbital. The energy of Fe^{3+} ions is small because its orbital angular moment is zero (Rada *et al.*, 2011)

From Zhang *et al.* (2007), cobalt oxide has a normal spinel structure and bulk. It is exhibits antiferomagnaetic with Neel temperature at around 30K. At the octahedral sites, the $Co_{3+}(3d_6)$ are diamagnetic in the octahedral crystal filed and at the tetrahedral sites, the Co_{2+} are form antiferromagnetic sub lattice with the diamond structure below the Neel temperature. From Shelby (2005) iron oxides, which are common impurities in the sands used to produce commercial silicate glasses. Its can act as unintentional colorants in many products.

Cobalt oxide with the formula Co_3O_4, is in organic compound. It has a molar mass 240.80gmol^{-1} with appearance in black solid. It's has melting point 895 °C and boiling point 900 °C. The density value of Co_3O_4 is 6.11gcm^{-3}. Iron oxide with molecular formula Fe_2O_3, is paramagnetic, reddish brown and readily attacked by acids. It has molar mass 159.69 $gmol^{-1}$ and density value 5.24 gcm^{-3}, with melting point 1566 °C.

1.5 Scope of study

A glass system containing transition metal oxide can be interesting material to study. Besides that, due to technological applications, the glass study doped with transition metal has been considerable interest study. This is because of their very sensitive responses to the surrounding cations and their outher d electron orbital function, it is used to probe the glass structure (Sreekanth Chakradhar, 2000). Because of that, the scopes of these studies are to produce new boro-tellurite glass system with adding cobalt oxide and iron oxide. In this work, the physical properties of glasses system has been determine. The dielectric properties in frequency range between 1.0^{-2}Hz to 1.0^{3}Hz of the glass system also have been analyzed such as ε' and ε'', conductivity,σ, modulus, M, and impedance plot, Z. As adding the cobalt oxide and iron oxide in the borotellurite glass system, it can give the magnetic properties behavior. For that, respond of the glasses sample with magnetic field also been investigated due to the magnetic properties behavior.

1.6 Problem statement

Due to the industrial important, boro-tellurite have been extensively studied. Tellurites is important in making glasses with desirable optical properties. Classical glass former B_2O_3 and conditional glass former TeO_2 present in boro-tellurite, leads complex specification and interesting properties in glass structure(Suresh *et al*., 2010). With introduction of transition metal such as Fe_2O_3 in glass system, which is a semiconducting glasses whose electrical properties and mechanism of the electronic conduction have been investigated extensively (Tanaka *et al*., 1990). Although many properties of boro-tellurite glass such as the silver borotellurite (Chowdari and Kumari, 1996), nickel borotellurite glasses(Khaled, 1992) and etc have attracted a number of researchers because of their wide range application, there is no systematic study of physical and dielectric properties of boro-tellurite glass doped with cobalt and iron oxide has been report. Therefore, an investigation of the physical and dielectric properties will be carried out and the results of this study are presented in this thesis.

1.7 Objectives

The objectives of this research as follows:

- To determine the physical properties of boro-tellurite glass system doped with cobalt oxide and iron oxide
- To study the dielectric behavior of the glass system such as dielectric permittivity, ε' and loss, ε'', conductivity, normalization curve (master plot), modulus, M and impedance plots, Z.
- To investigate the magnetic properties of the glasses system.

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