OPTICAL AND THERMAL PROPERTIES OF NEODYMIUM DOPED TELLURITE NANOSTRUCTURED GLASS

NUR AMANINA BINTI HJ MAT JAN

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I declare that this thesis entitled "*Optical and Thermal Properties of Neodymium Doped Tellurite Nanostructured Glass*" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : ……………………………

Name : NUR AMANINA BINTI HJ. MAT JAN

Date :

SPECIAL DEDICATED to

My Beloved Father and Mother

Hj. Mat Jan bin Hj. Abu Bakar

Hjh. Asiah bt Hj Hanapi

My Lovely Sisters

Nur Amirah

Nur Amalina

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ABSTRACT

Neodymium doped tellurite nanostructured glass having a composition of (75-x)TeO_2-15MgO-10Na_2O-(x)Nd_2O_3, where $0 \leq x \leq 2.5$ mol% has successfully been prepared by melt-quenching technique. The amorphous nature of the glass was confirmed from the X-ray diffraction pattern. Thermal parameter of the glass was analyzed by using differential thermal analysis. It was found that the glass exhibits the transition temperature between 255 - 259 ^{o}C, crystallization temperature between 428 to 450 ^{o}C and melting temperature from 505 to 549 ^{o}C. The nanoparticles were obtained by applying heat treatment to the glass at near crystallisation temperature of 460 ^{o}C for 30 minutes. The existence of nanocrystalline nature of this glass was confirmed by X-ray diffraction and transmission electron microscopy. The vibrational study was conducted using Fourier transform infrared spectroscopy in the range 4000 - 400 cm^{-1}. It was found that there were three absorption peaks around 687 - 712 cm^{-1}, 624 - 632 cm^{-1} and 467 - 474 cm^{-1} which are due to the stretching of Te-O bending in TeO_3 trigonal pyramidal (tp) units, Te-O bands stretching vibration in TeO_4 trigonal bipyramidal (tbp) units and the bending vibrations of Te-O-Te or O-Te-O linkages, respectively. The optical absorption behaviour was studied using UV-Visible spectrophotometer. The values of indirect optical band gap are in the range 3.15 - 3.22 eV and for direct band gap the values are in the range 3.37 - 3.43 eV. The Urbach energy value for glass system was found to be in range 0.208 eV and 0.125 eV. The emission spectrum was recorded using photoluminescence spectrometer. The fluorescence spectra of Nd^{3+} ions exhibit emission transition of $^2P_{1/2} \rightarrow {}^4I_{9/2}$, $^2D_{3/2} \rightarrow {}^4I_{9/2}$ and $^4G_{7/2} \rightarrow {}^4I_{9/2}$ under 585 nm excitation wavelengths. It was found that the intensity is enhanced in the heat-treated glass compared to those of prepared glass.

ABSTRAK

Kaca tellurit berstruktur nano didopkan neodimium yang mempunyai komposisi (75-x)TeO_2-15MgO-10Na_2O-(x)Nd_2O_3, dengan $0 \leq x \leq 2.5$ mol% telah berjaya disediakan menggunakan teknik pelindapan leburan. Sifat amorfus kaca ditentukan daripada corak pembelauan sinar-X. Parameter terma kaca dianalisis menggunakan analisis perbezaan terma. Kaca ini didapati mempamerkan suhu peralihan antara 255 - 259 ^oC, suhu penghabluran kaca antara 428 - 450 ^oC dan suhu lebur kaca daripada 505 ke 549 ^oC. Zarah nano diperoleh dengan melakukan rawatan haba ke atas kaca pada suhu hampir dengan suhu penghabluran kaca pada 460 ^oC selama 30 minit. Kewujudan nanohablur kaca disahkan dengan pembelauan sinar-X dan mikroskopi penghantaran elektron. Kajian terhadap getaran telah dilakukan dengan menggunakan spektroskopi inframerah transformasi Fourier (FTIR) dalam julat 4000 - 400 cm^{-1}. Tiga puncak penyerapan yang ketara terdapat sekitar 687 - 712 cm^{-1}, 624 - 632 cm^{-1} dan 467 - 474 cm^{-1} masing-masing merujuk kepada regangan bengkokan Te-O dalam unit TeO_3 *piramid trigonal* (tp), getaran regangan jalur Te-O dalam unit TeO_4 *bipiramid trigonal* (tbp) dan getaran bengkokan Te-O-Te atau O-Te-O. Sifat serapan optik dikaji menggunakan spektrofotokopi ultralembayung-cahaya nampak. Nilai tenaga jurang optik tidak langsung ialah antara 3.15 - 3.22 eV dan tenaga jurang optik langsung ialah antara 3.37 - 3.43 eV. Tenaga Urbach bagi kaca adalah dalam julat 0.208 eV dan 0.125 eV. Spektrum pancaran telah direkod menggunakan spektrometer fotoluminesen. Spektra pendarcahaya Nd^{3+} menunjukkan peralihan pancaran $^2P_{1/2} \rightarrow {}^4I_{9/2}$, $^2D_{3/2} \rightarrow {}^4I_{9/2}$ dan $^4G_{7/2} \rightarrow {}^4I_{9/2}$ pada pengujaan 585 nm. Keamatan kaca rawatan haba didapati menunjukkan peningkatan keamatan berbanding kaca yang tiada rawatan haba.

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LIST OF SYMBOLS

TeO_2	-	Tellurite oxide
Na_2O	-	Sodium oxide
PbO	-	Lead oxide
BaO	-	Barium Oxide
ZnO	-	Zinc Oxide
Nb_2O_5	-	Niobimium Oxide
WO_3	-	Tungsten trioxide
$Te\text{-}O_{ax}$	-	Te-O axial
Li_2O	-	Lithium Oxide
Mg	-	Magnesium
Zn	-	Zinc
Nd^{3+}	-	Trivalent Neodymium ion
OH^-	-	OH ion

SiO_2	-	Silicon Dioxide
GeO_2	-	Germanium Dioxide
P_2O_5	-	Phosphorus Pentoxide
B_2O_3	-	Boron Trioxide
$Al(PO_3)$	-	Aluminium Phosphate
$Ba(PO_3)$	-	Barium Phosphate
KH_2PO_4	-	Pottasium Phosphate
$Mg(PO_3)_2$	-	Magnesium Phosphate
Cr^{6+}	-	Chromium (VI) ion
NBO	-	Non-bridging oxygen
NPs	-	Nanoparticles
α_m	-	Molar polarizability
α_0^{2-}	-	Electric polarizability
Λ_{th}	-	Theoritical basicity
(N/V)	-	number of ions per unit volume
tp	-	trigonal pyramidal

tbp	-	trigonal bipyramidal
λ	-	Wavelength
E_a	-	Activation energy
$E_{direct\ opt}$	-	Optical direct band gap energy
$E_{indirect\ opt}$	-	Optical indirect band gap energy
E_{opt}	-	Optical band gap energy
ΔE_f	-	Free energy
ΔE	-	Urbach energy
E_F	-	Fermi energy level
U	-	Growth rate
T_d	-	Development temperature
T_n	-	Nucleated temperature
T_m	-	Melting temperature
T_c	-	Crystallization temperature
T_g	-	Glass transition temperature
H_R	-	Hruby criterion
S	-	Glass stability

v_{as}	-	Asymmetric stretching vibration
v_s	-	Symmetric stretching vibration
D	-	Crystal Size
θ	-	Bragg Angle
β	-	FWHM
I_0	-	Reference beam
I	-	Sample beam
A	-	Absorbance
T	-	Transmittance
$\alpha(\omega)$	-	Absorption coefficient
$\hbar\omega$	-	Photon energy

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CHAPTER 1

BACKGROUND

1.1 Introduction

In this chapter, the background, problem statement and the objective of this study will describe in details. The scope and the significant of this study will also be touch briefly.

1.2 Background of Study

Tellurite glasses have several advantages compared to silica, fluoride and chalcogenide glasses. They have wider transmission region (0.35 - 5 μm) than silicate glasses (0.2 - 3 μm). Tellurite glasses exhibit high non-linear refractive index compare to those of fluoride, phosphate and silicate glasses relatively.

They also have good glass stability and highly corrosion resistance compare to those of fluoride glasses. Their lower melting temperature and lower phonon energy make them attractive hosts for active medium. TeO_2-based glasses with lower phonon energy could have higher quantum efficiencies and provide more fluorescent emissions than silica-based glasses. This implies that the tellurite glasses are suitable for nonlinear and laser applications.

Other than that, tellurite glasses are of interest due to better chemical durability and higher glass stability than halide glasses (Stanworth, 1962), and better compatibility when mix with other oxide glasses than the nonoxide glasses. From the data given in Table 1.1, it can be seen that the degree of covalence among the four glass family decreases in order of Chalcogenide > Tellurite > Silica > Fluoride glasses, which directly affects the wavelength of the transition peak (Wang *et al.*,1994).

Table 1.1: A comparison of selected properties for tellurite, silica, fluoride and chalcogenide glasses (Wang *et al.*,1994).

Property	Tellurite	Silica	Fluoride	Chalcogenide
Optical properties (typical values)				
Refractive index (n)	1.8–2.3	1.46	1.5	2.83
Abbe number (ν)	10–20	80	60–110	
Nonlinear refractive index (n_2, m^2/W)	2.5×10^{-19}	10^{-20}	10^{-21}	higher
Transmission range (μm)	0.4–5.0	0.2–2.5	0.2–7.0	0.8–16
Highest phonon energy (cm^{-1})	800	1000	500	300
Longest fluorescent wavelength (μm)	2.8	2.2	4.4	7.4
Bandgap (eV)	≈3	≈10		1–3
Acousto-optical figure of merit, $p^2n^6/r v^3$ (10^{-18} s^3/g)	24	1–19	–	
Physical properties (typical values)				
Glass transition (T_g, °C)	300	1000	300	300
Thermal expansion (10^{-7} °C)	120-170	5	150	140
Density (g/cm^3)	5.5	2.2	5.0	4.51
Dielectric constant (ϵ)	13–35	4.0	–	
Fiber loss	–	0.2 dB/km (1.5 μm)	15–25 dB/km (1.5–2.75 μm)	0.4 dB/km 6.5 μm
Bonding	covalent-ionic	ionic-covalent	ionic	covalent
Solubility in water	$<10^{-2}$	$<10^{-3}$	soluble	$<10^{-4}$

Since TeO_2 does not form glass by itself, it belongs to the intermediate class of glass-forming oxides usually, during glass preparation, metal oxides modifier such as Na_2O, PbO, BaO, ZnO, Nb_2O_5, WO_3 are added to it. Modifier molecules usually enhance the glass formation ability (GFA) in glass formers by breaking chains of structural units. It will cause changes in structural formation units (Stanworth, 1952; Suehara *et al.*, 1995; El-Mallawany, 2002; Narayanan and Zwanzinger, 2003). The structure of TeO_2 rich glasses have trigonal bipyramidal TeO_4, deformed TeO_4, TeO_{3+1} polyhedron and trigonal TeO_3 structural units. The network modifiers atoms can be easily attack two highly mobile axial bonds (Te-O_{ax}) in each TeO_4 unit. One of the Te-O_{ax} bonds in TeO_4 polyhedra elongates, the bond length increases and forms TeO_{3+1} structural unit when a network modifier like metal oxide is added into the glass matrix. Kaur *et al.* reported that the subscript 3+1 indicates that the fourth oxygen is nearby but is not within a true bonding range. Structural unit of TeO_3 may be defined when the Te-O bond length exceeds the average length (Singh, 1997). As for notation, TeO_4 can be labeled as Q_4^4, TeO_{3+1} as Q_4^3, and TeO_3 as $Q_3^{2,1,0}$ where the subscript represents the coordination number of oxygen around Te atom and the superscript is the number of bridging oxygens linked to a Te atom (Kaur *et al.*, 2010). However, this notation is not commonly been used except for phosphate glasses.

Modifier such as Na_2O or Li_2O is necessary to add to TeO_2 or otherwise the melted material quickly recrystalized. It is reported that the modifying elements such as Mg, Zn, and Ba are chosen for their ability to aid in the formation of stable tellurite glasses (Nishida *et al.*, 1990). The concentration of about 10 mol% of sodium is necessary to increase the glass forming stability of the mixtures is proven by Mc Laughlin in 2000. The combination of MgO with TeO_2 for example, will increase the glass transition temperature because of Mg-O bonds have much higher polarity than the Zn-O bonds due to the lower electronegative. Therefore, these bonds require energy to break. The existence polar bonds into the glass will result in the increase of glass the transition temperature (Sean Manning *et al.*, 2011). Other usage of those glasses includes potential application in pressure sensors or new laser host because of excellent infrared transmission (0.4 - 6.0 µm) (Singh *et al.*, 2007; Burger *et al.*, 1992).

Recently tellurite glasses doped with heavy metal and rare earth oxides have received significant attention because they can favorably change density, optical and thermal properties of tellurite glasses (Kaur *et al.*,2010). Tellurite glasses also promising applications for upconversion of infrared to visible light, ultra broadband fiber Raman amplifiers (Murugan *et al.*, 2005) and gas sensors (Chakraborty *et al.*, 1997).

Neodymium doped all-solid-state laser sources have been identified as the most efficient laser sources for numerous applications in the fields of high-resolution spectroscopy. The development of low threshold high gain host media for Nd^{3+} ion doping is encouraged by the applications in these areas. It is obvious that the enlargement of host material for Nd^{3+} ions requires optimum material properties. They are characterized by a low content of OH^- groups and a low frequency phonon spectrum. The low in OH^-content make it possible for the glass to reduce excitation losses due to the multiphonon relaxation. In addition, the OH^- free tellurite glasses which are used as host material for Nd^{3+}-doped laser glasses have been a subject of increasing interest for optoelectronic applications. It is because of their high refractive index and low phonon energies (Rajeswari, 2010).

According to Bogdanov, doping with larger rare earth ions concentration is need high pumping absorption in a short cavity length (Bogdanov, 1996). Then, Santa-Cruz *et al.* (1995) had further to this method to produce a glass system in which the local crystal field environment of the rare earth ions is the same as in the crystalline form. However, the homogeneity of the glass reduced by the formation of grain boundary thus producing some losses during pumping process (Santa-Cruz *et al.*, 1995).

1.3 Problem Statement

These nanostructered glasses have much technological interest especially because of their thermodynamic properties which show strong size dependence and can easily controlled by manufacturing processes. Lasing properties and capabilities of the glass can be made by controlling their structural development up to nanoscale. However, there is lack of information in the literature about the properties of Nd_2O_3 doped tellurite nanostructered glass. Furthermore, the method of producing the nanocrystal in the glass matrix using heat treatment is not well reported. Therefore, the aim of this study is to characterize the optical and thermal properties of Nd_2O_3 doped tellurite nanostructered glass.

1.4 Objective of Study

The objectives of this research are:

i. To prepare the glass based on $(75-x)TeO_2-15MgO-10Na_2O-(x)Nd_2O_3$ by using melt quenching technique.
ii. To determine the crystallization temperature of Nd^{3+} doped tellurite using Differential Thermal Analysis (DTA).
iii. To heat-treated the glass and determine the size of nanoparticles in the prepared sample using X-Ray Diffraction (XRD) and Transmission Electron Microscopy (TEM).
iv. To characterize the transmission behavior by using Fourier Transform Infra red (FTIR) spectroscopy.
v. To determine the optical band gap and Urbach energy by using UV-Vis spectroscopy.
vi. To characterize the emission properties by using photoluminescence spectroscopy.

1.5 Scope of Study

In sequence to achieve the above objectives the works have been focus on the preparation of glass based on the (75-x)TeO_2-15MgO-10Na_2O-(x)Nd_2O_3 system ($0 \leq x \leq 2.5$ mol%) using the melt quenching technique. The amorphous or crystalline phase present in the prepared sample will be identified. The thermal properties of the samples will be measured by using Differential Thermal Analysis (DTA). The feature of nanocrystalline phase will be observed by x-ray diffraction (XRD) and Transmission Electron Microscopy (TEM). The transmission spectra will be characterized by using Fourier Transform infrared (FTIR) spectrometer. The value of optical band gap and Urbach energy will be determined by using UV-VIS spectrophotometer. The emission of prepared glass will be characterized by using Photoluminescence Spectrometer.

1.6 Significant of Study

The reason why this study is being conducted is to find out the optical and thermal properties of TeO_2-MgO-Na_2O nanostructured glass which is very promising development in optical technological applications. The obtained data can be used to enhance the glass capability to increase the possibility of the glass in laser emission.

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