SYNTHESIS AND CHARACTERIZATIONS OF DYSPROSIUM DOPED ZINC-SODIUM-TELLURITE GLASS

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To

my parents and siblings, whose sacrifice, dream, support and encouragement lead to achieve my present educational level.

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ABSTRACT

Rare earth ions (REIs) doped binary and ternary tellurite glass systems are attractive because of several technological appications. Transparent tellurite glasses can be achieved by combining the vitrified tellurium oxide (TeO_2) host with various modifier oxides of Zinc (ZnO), Sodium (Na_2O), Silicon (SiO_2), etc. over a wide composition range. Tellurite system is wellknown for large intake of REIs, excellent optical properties and low phonon energy cutoff. Determining the influence of dysprosium (Dy^{3+}) ions doping on the improvement of physical, structural and optical properties of tellurite glasses is the main focus of this research. A series of Dy^{3+} ions doped zinc-sodium-tellurite glass having composition of (65-x)TeO_2-25ZnO-10Na_2O-xDy_2O_3 ($0 \leq x \leq 2.5$ mol%) are prepared using melt-quenching method. Synthesized samples are characterized at room temperature via different analytical techniques. X-ray diffraction pattern verified the amorphous nature of the synthesized glass samples in the absence of any sharp crystalization peaks together with the presence of a braod hump between 25°-35° diffraction angle. Differential thermal analysis revealed good thermal stability of the glass system in the range of 120-206 ℃ and Hruby's parameter between 0.47-1.33. Dy^{3+} ions concentration dependent density and molar volume of the glass system is found vary in the range of 5.334 gcm^{-3} - 5.366 gcm^{-3} and 24.425 cm^3mol^{-1} - 25.273 cm^3mol^{-1}, respectively. Fourier transformed infrared spectra exhibited bonding vibrations at the wavenumber of 590-615 cm^{-1} and 772-817 cm^{-1} which are assigned to [TeO_4] and [TeO_3] glass network structural units, respectively. Ultraviolet-Visible-Near-Infrared (UV-Vis-NIR) spectra displayed seven absorption peaks centred at 450, 752, 801, 901, 1095, 1281, and 1687 nm which are allocated to the transitions from the ground level to the excited levels (such as $^4F_{9/2}$, $^6F_{3/2}$, $^6F_{5/2}$, $^6F_{7/2}$, $^6H_{7/2}$, $^6F_{11/2}$, and $^6H_{11/2}$) of Dy^{3+} ions, respectively. The Dy^{3+} ions contents dependent UV-Vis absorption edge data is used to calculate various optical properties of the glass. Indirect optical band gap is decreased from 2.67-2.30 eV and the Urbach energy is increased from 0.265-0.421 eV with increasing concentration of Dy^{3+} ions. This indicated the enhancement of glass compactness and structural change mediated via non-bridging oxygen atoms in the network. Room temperature photoluminescence (PL) spectra showed three significant peaks centred at 497, 588, and 675 nm, which are attributed to the transitions from $^4F_{9/2}$ excited state to the $^6H_{11/2}$, $^6H_{13/2}$, and $^6H_{15/2}$ states of Dy^{3+} ions, respectively. Highest PL intensity enhancement of 1.54 and 1.63 for yellow and red band, respectively is obtained for glass samples with 0.8 mol% Dy^{3+} ions. The blue band of the sample with 1.2 mol% Dy^{3+} ions revealed 1.46 times PL enhancement. This enhancement is ascribed to the excited state absorption and cross-relaxation processes. Overall, it is demonstrated that the physical, structural, thermal, and optical properties of the tellurite glass is improved due to the incorporation of Dy^{3+} ions in the host matrix. Present optimized glass composition may be potential for the development of solid-state lasers and other photonic devices.

ABSTRAK

Sistem kaca telurit binari dan ternari dop ion nadir bumi atau *Rare Earth Ions* (REIs) adalah menarik berikutan beberapa aplikasi teknologi. Kaca telurit telus boleh dihasilkan dengan menggabungkan perumah kekaca telurium oksida (TeO_2) dengan pelbagai oksida pengubahsuai Zink (ZnO), Natrium (Na_2O), Silikon (SiO_2), dan lain-lain dalam julat komposisi yang luas. Sistem Telurit diketahui umum kerana pengambilan REI yang tinggi, ciri optik yang sangat baik dan penggalan tenaga fonon rendah. Menentukan pengaruh pengedopan ion disprosium (Dy^{3+}) terhadap peningkatan ciri fizikal, struktur dan optik kaca telurit ialah fokus utama kajian ini. Satu siri kaca zink-natrium-telurit dop ion Dy^{3+} dengan komposisi (65-x) TeO_2-25ZnO-10Na_2O-xDy_2O_3 ($0 \leq x \leq 2.5$ mol%) disediakan mengikut kaedah leburan-pelindapkejutan. Sampel disintesis dicirikan pada suhu bilik melalui teknik analisis berbeza. Pola pembelauan sinar-X mengesahkan sifat amorfus sampel kaca disintesis tanpa sebarang puncak penghabluran tajam, bersama-sama dengan kehadiran bonggol lebar dengan sudut pembelauan antara 25°-35°. Analisis terma berbeza mendedahkan kestabilan terma sistem kaca yang baik pada julat 120-206℃ dan parameter Hruby antara 0.47-1.33. Ketumpatan bersandarkan kepekatan ion Dy^{3+} dan isi padu molar sistem kaca itu didapati berbeza-beza, masing-masing pada julat 5.334 gcm^{-3}-5.366 gcm^{-3} dan 24.425 cm^3mol^{-1}-25.273 cm^3mol^{-1}. Spektrum inframerah terjelma Fourier menunjukkan getaran ikatan pada nombor gelombang 590-615 cm^{-1} dan 772-817 cm^{-1} yang diberikan, masing-masing, kepada unit struktur rangkaian kaca [TeO_4] dan [TeO_3]. Spektrum *Ultraviolet-Visible -Near-Infrared* (UV-Vis-NIR) memaparkan tujuh puncak penyerapan berpusat di 450, 752, 801, 901, 1095, 1281, dan 1687 nm yang diperuntukkan kepada peralihan dari aras bawah ke aras teruja (seperti $^4F_{9/2}$, $^6F_{3/2}$, $^6F_{5/2}$, $^6F_{7/2}$, $^6H_{7/2}$, $^6F_{11/2}$, dan $^6H_{11/2}$) ion Dy^{3+}, masing-masing. Data tepi penyerapan UV-Vis bersandarkan kandungan ion Dy^{3+} digunakan untuk mengira pelbagai ciri optik kaca. Jurang jalur optik tidak langsung menurun daripada 2.67-2.30 eV dan tenaga Urbach bertambah daripada 0.265-0.421 eV dengan peningkatan kepekatan ion Dy^{3+}. Ini menunjukkan peningkatan kepadatan kaca dan perubahan struktur melalui pengantaraan atom oksigen bukan penitian dalam rangkaian. Spektrum kefotopendarcahayaan (PL) suhu bilik menunjukkan tiga puncak yang ketara berpusat di 497, 588, dan 675 nm, yang disebabkan oleh peralihan dari keadaan teruja $^4F_{9/2}$ kepada keadaan $^6H_{11/2}$, $^6H_{13/2}$, and $^6H_{15/2}$ ion Dy^{3+}, masing-masing. Peningkatan intensiti PL tertinggi pada 1.54 dan 1.63 untuk jalur kuning dan merah, masing-masing diperolehi dengan sampel kaca pada 0.8mol % ion Dy^{3+}. Jalur biru sampel dengan 1.2mol% ion Dy^{3+} mendedahkan 1.46 kali peningkatan PL. Peningkatan ini dianggap berpunca daripada proses penyerapan keadaan teruja dan pengenduran silang. Secara keseluruhan, didapati bahawa sifat-sifat fizikal, struktur dan optik kaca telurit bertambah baik disebabkan oleh penggabungan ion Dy^{3+} dalam matriks perumah. Komposisi kaca teroptimum sekarang mungkin berpotensi untuk pembangunan laser keadaan pepejal dan peranti fotonik lain.

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LIST OF ABBREVIATIONS

BS	-	Beam Splitter
BOs	-	Bridging Oxygens
CN	-	Coordination Number
CR	-	Cross Relaxation
DC	-	Down Conversion
DTA	-	Differential Thermal Analyser
ET	-	Energy Transfer
ENDO	-	Endothermic
EXO	-	Exothermic
EDX	-	Energy Dispersive Electron Microscope
FIR	-	Far Infrared
FTIR	-	Fourier Transform Infrared
hcp		Hexagonal Closed-Packed
HSTs	-	High Sensitive Transitions
IR	-	Infrared
MIR	-	Mid Infrared
NBO	-	Nonbridging Oxygen
NIR	-	Near Infrared
NMR	-	Nuclear Magnetic Resonance
NR	-	Nonradiative
PL	-	Photoluminescence
RE	-	Rear Earth
REIs	-	Rear Earth Ions
tbp	-	Trigonal Bipyramid
tp	-	Trigonal Pyramid
UP	-	Up Conversion
UV-Vis-NIR	-	Ultraviolet-Visible-Near Infrared

VB	-	Valence Band
XRD	-	X-Ray Diffraction

LIST OF SYMBOLS

2θ	-	Diffraction angle
α_e	-	Polarizability
$\alpha(\nu)$	-	Absorption coefficient
$\AA$	-	Angstrom
Ax	-	Axial
℃	-	Degrees centigrade
D	-	Crystal lattice planar spacing
ΔE	-	Urbach energy
E_c	-	Conduction band energy
E_{opt}	-	Optical band gap energy
Eq	-	Equatorial
E_v	-	Valence band energy
H	-	Hruby parameter
$H\nu$	-	Photon energy
$I_{sample}(q)$	-	Luminescence intensity of other glass samples
$I_{reference}$	-	Luminescence intensity of glass sample with 0.4 mol% of Dy^{3+} ion
K	-	Kelvin
KBr	-	Potassium bromide
Ln	-	Lanthanide
M_{av}	-	Average molecular weight
N	-	Refractive index of glass
N_A	-	Avogadro's number
$N(E_c)$	-	Density of states at the conduction band
$N(E_v)$	-	Density of states at the valence band
n_r	-	Order of reflection

Θ	-	Angle of incidence
σ_o	-	Conductivity
Λ	-	Wavelength
ρ	-	Glass density
ρ_a	-	Air density
ρ_l	-	Density of immersion liquid
R	-	Optical transition index
R_m	-	Molar refractivity
T	-	Temperature
T_c	-	Glass crystallization temperature
T_g	-	Glass transition temperature
T_m	-	Glass melting temperature
ΔT_s	-	Glass thermal stability
T_x	-	Glass onset crystallization temperature
N	-	Frequency
V_m	-	Molar volume
W_a	-	Weight of glass in air
W_l	-	Weight of glass in immersion liquid

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CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter gives a brief background on the development of glass research especially tellurite glass, aims towards potential application in solid state physics and other optical devices. Also it introduce the problem statement, objectives, scope of the study and significamce of the research.

1.2 Research Background

In the realm of controlled modified and enhance properties of rare earth (RE) doped glass for various practical application, achieving this, is a quest which cannot be over-emphasised. Having special properties in relation to plastic or metal, makes glass a unique material. Some of this properties makes it to be increasingly used as good host for laser, based on RE and metal ionic transitions. Tellurite based glasses are regarded as excellent materials for the above purpose (host for lasing ions). Conversely, tellurium dioxide (TeO_2) as a pure oxide, manifests itself as simply a restrictive glass-former, which needs a special fast-quenching technique to vitrify [1]. Vitrifying TeO_2 using traditional process encountered difficulty, achieving high transparent tellurite glasses are done by hosting other oxides like transition metal oxides: Zinc oxide (ZnO), alkaline oxides: sodium oxide (Na_2O) and alkaline earth oxide: magnesium oxide (MgO) devoid of adding any conventional glass former such as P_2O_5, SiO_2 and B_2O_3 [2]. Physical, structural and optical parameters of rare earth ions (REIs) doped glasses have been extensively examined owing to their prospective uses in fabrication of innovative optical devices. As uncover via optical studies, the radiative behaviour of the REIs in glasses is intensely influenced by the host matrix, also, can be improved through suitable selection of network former together with network modifier ion [3].

Over the years RE predominantly Dy^{3+}, Eu^{3+}, Sm^{3+}, Er^{3+}, and Pr^{3+}, doped glasses shown to be eye-catching in emerging countless active optical devices [4]. Numerous researches worked on Dysprosium (Dy^{3+}) ion owing to it prospect in fabrication of various types of light emitting materials for blue, yellow and red luminescence conforming to transitions of $^4F_{9/2} \rightarrow {}^6H_J$, (J=15/2, 13/2, 11/2) respectively [5, 6].

1.3 Motivation of the Study

Owing to its optical absorption, glass displays certain colours, which have been intensively utilised in the areas of optical fibre communications and optical switching [7]. A number of compositions of glasses are being synthesised, with enormous properties by means of different families of the periodic table such as oxide, chalcogenide, halide etc. [1].

Oxide glasses are synthesised from a number of oxides commonly SiO_2, P_2O_5, B_2O_3, GeO_2, TeO_2 etc. One of the advantages of oxide glasses is that they can be simply prepared by melt quench technique, with challenging properties paralleled to chalcogenide glasses [1].

Among the oxide glasses one of the most proper hosts for doping RE element are thought to be tellurite glasses, making it to have growing attention due to its unique physical properties. In latest years, significant consideration has been given to solid materials doped with RE owing to their prospective usage as colour displays, glowing and optoelectronic devices [8, 9].

The REIs energy levels decide the lasing properties of RE doped systems and are subjective to glass former matrix [10]. The optical characteristics of zinc flurophosphate glasses doped with Dy^{3+} ions were described [11]. Sasi kumar *et al*, 2013 put forward the absorption and PL behaviour of Dy^{3+}-doped heavy metal borate glasses-effect of modifier oxides [12]. Swapna, *et al*, 2013 explored the absorption and luminescence properties of Dy^{3+}-doped zinc alumino bismuth borate glasses for laser devices so also white light emitting diodes (LEDs) [13]. Balakrishna, *et al*, 2012 considered structural and PL characteristics of Dy^{3+} doped different modifier oxide-based lithium borate glasses [14].

1.4 Problem Statement

There have been some limitations of REIs doped tellurite glass due to low absorption and emission cross-section (measure for the probability of absorption and emission occurrance) in spite of various distinct features [15]. However, the low stability, low absorption and emission as well as structural strength of tellurite glass needs improvement. In this regard, optimum composition of tellutite glass system is not been explored yet [16]. Discovering new prospective lasing transition and to raise the component of lasing transitions, REIs doped glass has been painstakingly investigated in diverse glass base, so also in particular their optical behaviour [17].

Much concern on tellurite glasses are motivated by their use in several industrial usages specifically in laser glass technology [18]. Dy^{3+} ions are doped inside the tellurite glass system because of their sharp emission line in the IR and visible region. Moreover, tellurite glass is a good host that produce enhanced absorption and emission properties of rare earth suitable for laser application [19]. Yet most of the report are partly on tellurite glasses doped with Eu^{3+}, Nd^{3+} and Er^{3+}.

Not much studies have been reported on the Dy^{3+} doped zinc-sodium-tellurite glasses and also the optimum concentration with this rare earth ion is not develop yet.

In this study, it shown synthesis and characterization of zinc sodium tellurite glass doped with Dy^{3+}. Also low transition temperature and presence of hygroscopic properties limit the application of phosphate and borate glasses in comparism to zinc sodium tellurite glass system [20]. The comprehensive information on the structures, thermal and optical properties of Dy^{3+} doped zinc-sodium-tellurite glasses are still deficient [21]. Therefore structural thermal and optical properties of the glass system under study need to be investigated.

1.5 Objectives of the Study

The objective are listed below:

i. To synthesize Dy^{3+} ions doped zinc sodium tellurite glass with optimum composition.
ii. To characterize the prepared Dy^{3+}-doped zinc-sodium tellurite glass systems for their physical, structural and thermal properties.
iii. To determine the influence of Dy^{3+} ions on the optical properties of the synthesized glass systems.

1.6 Scope of the Study

Owing to the utmost result of TeO_2-ZnO-Na_2O glass system as put forward by [22-24] leading to the decision of (65-x)TeO_2-25ZnO-10Na_2O-xDy_2O_3 as the preferred composition for this study. As reported heavy metal oxide (like ZnO) has been used as a favourable constituent materials aimed for tellurite- based fibres [25] The study of structural, thermal and optical properties of glasses is crucial to give a clear understanding of the growth and thermal stability which is fundamental to prepare a glass with optimal properties.

i. The amorphous nature of the glass was comfirmed using XRD.

ii. Further study on structural behaviour as regard to bonding vibrations of the glasses are demonstrated by FTIR spectroscopy.

iii. The thermal properties of glass including the crystallization temperature, glass transition temperature, glass stability and Hruby's parameter are determined by using differential thermal analysis (DTA).

iv. Meanwhile, the optical properties of glass are studied by using UV-Vis-NIR and PL spectroscopy.

The thermal, optical and structural studies are vital to select suitable glass with optimized parameters to be used in various optical-based devices [26]. This study gives an in-depth on aformentioned spectroscopic properties of zinc-sodium tellurite glass system doped with Dy^{3+} ions.

1.7 Significance of the Study

The search for profitable and industrial down-converted lasing glass is eternally swelling. Due to the demand for several industrial applications, more study on glass materials is vital. There is need to relate in many aspects such as physical, optical, structural and other properties with precise and superior characteristics manufactured glassy materials.

i. This dissertation will allows us to synthesize a glass composition by doping Dy^{3+} ion, The improvement in the structural, optical, thermal and physical properties will be understood by systematic characterizations.

ii. Thermal stability will be improved.

iii. The enhancement of the absortion and emission properties of the glass will be useful for many optical devices.

iv. The mechanism of optical enhancement and structural change due to the influence of Dy^{3+} ion will be uderstood clearly.

1.8 Thesis Outline

Chapter one comprises the reserch background of tellurite glass, discusses the importance of the samples to be prepared in relation to its optical, photoluminescence, thermal and structural properties. Zinc-sodium-tellurite glass doped with dysprosium is to present a glass material with potential in optical devices. The set objectives to be achieved are also enumerated.

Chapter two, it offered wide-ranging explanation concerning of glass structure and behavour of Zinc, Sodium as modifiers to tellurite sytems with the influence of dysprosium as RE dopant. The physical, optical, thermal and structural properties likewise will be reviewed.

Chapter three, it encompases the procedural steps used in the glass preparation, techniques employed in investigating the various spectroscopic measurements with the physical properties are well detailed.

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