

OPTICAL PROPERTIES OF SAMARIUM AND YTTERBIUM DOPED SODIUM
TELLURITE GLASSES

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ABSTRACT

Interests in rare earth doped/co-doped inorganic binary lasing glasses with enhanced efficiency are ever-growing. Optimizing the rare earth concentrations to achieve significant optical performance is becoming more challenging. Two series of $\text{Sm}^{3+}/\text{Yb}^{3+}$ co-doped sodium tellurite glasses of composition $(79.5-x)\text{TeO}_2-20\text{Na}_2\text{O}-x\text{Sm}_2\text{O}_3-0.5\text{Yb}_2\text{O}_3$ with $0 \leq x \leq 2.0$ mol% and $(79.5-y)\text{TeO}_2-20\text{Na}_2\text{O}-0.5\text{Sm}_2\text{O}_3-y\text{Yb}_2\text{O}_3$ with $0 \leq y \leq 2.0$ mol% were synthesized using melt quenching technique. The influence of co-dopants on structural and optical properties of these glasses was determined. XRD pattern confirmed the amorphous nature of the glasses. Density, molar volume and the refractive index were found to be highly influenced by co-dopants. The temperature for glass transition, crystallization and melting were determined from DTA thermogram. The observed thermal stability in the range of 104 to 232 °C verified wide and stable range of glass formation. FTIR spectra revealed different modes of vibration with increasing transmission bands shift from 616 to 651 cm^{-1} which corresponds to stretching vibration mode of TeO_4 . Conversely, the Raman shift in intensity was found to decrease around 681-690 cm^{-1} and increase around 756-758 cm^{-1} due to the change in composition. These observations are ascribed to the structural changes between the stretching vibration mode of TeO_4 trigonal bipyramid and TeO_3 trigonal pyramid and the bending vibration mode within the Te-O-Te linkages through the creation of non-bridging oxygen atoms. The sizable alteration in the structure is evidenced due to co-dopants. The UV-VIS-NIR absorption spectra display eight prominent absorption peaks centered at 470, 948, 1086, 1238, 1386, 1492, 1550 and 1588 nm corresponding to transitions from ground state to various excited states. The absorption data was further complemented with the calculation of Judd-Ofelt intensity parameters Ω_2 , Ω_4 and Ω_6 . The variation in Ω_2 showed covalent nature of bonding and the spectroscopic quality factor was found to be around 171-251. The room temperature emission spectra for both glasses exhibited four peaks with enhanced intensity up to certain concentrations of co-dopants and quenching thereafter which was understood using partial energy level diagram. The maximum values of stimulated emission cross-section, gain band width and optical gain for ${}^4\text{G}_{5/2} \rightarrow {}^6\text{H}_{9/2}$ transitions were observed to be $23.09 \times 10^{-22} \text{ cm}^2$, $28.54 \times 10^{-28} \text{ cm}^3$ and $12.51 \text{ cm}^2 \text{ s}^{-1}$, respectively. A correlation was established between the $\text{Sm}^{3+}/\text{Yb}^{3+}$ co-doping and modification in structural and optical properties of the studied glasses. The excellent features of these results suggest that these glasses may be nominated as potential candidate for solid state lasers and other photonic devices.

ABSTRAK

Minat terhadap kaca laser binari tak organik berkecekan tinggi yang didop/ ko-dop dengan unsur nadir bumi semakin meningkat. Pengoptimuman kepekatan unsur nadir bumi bagi mendapat prestasi optik yang signifikan adalah sangat mencabar. Dua siri kaca natrium tellurit yang diko-dop dengan $\text{Sm}^{3+}/\text{Yb}^{3+}$ berasaskan komposisi $(79.5-x)\text{TeO}_2-20\text{Na}_2\text{O}-x\text{Sm}_2\text{O}_3-0.5\text{Yb}_2\text{O}_3$ dengan $0 \leq x \leq 2.0$ mol% dan $(79.5-y)\text{TeO}_2-20\text{Na}_2\text{O}-0.5\text{Sm}_2\text{O}_3-y\text{Yb}_2\text{O}_3$ dengan $0 \leq y \leq 2.0$ mol% telah disediakan melalui teknik pelindapan leburan. Pengaruh ko-pondop ke atas struktur dan sifat optik kaca tersebut telah ditentukan. Corak belauan XRD telah menentusahkan bahawa kaca adalah amorfus. Ketumpatan, isipadu molar dan indeks biasan didapati sangat bergantung kepada bahan ko-pondop. Suhu transisi, suhu penghabluran dan suhu peleburan kaca telah ditentukan melalui termogram DTA. Kestabilan terma kaca didapati berada dalam julat 104 °C hingga 232 °C, menunjukkan julat pembentukan kaca yang lebar dan stabil. Spektra FTIR menunjukkan mod getaran yang berbeza dengan anjakan jalur kehantaran yang meningkat daripada 616 cm^{-1} ke 651 cm^{-1} yang menunjukkan mod regangan getaran TeO_4 . Sebaliknya, anjakan keamatan Raman di sekitar 681-690 cm^{-1} didapati mengurang manakala di sekitar 756-758 cm^{-1} pula meningkat disebabkan perubahan komposisi kaca. Pemerhatian ini adalah disebabkan oleh perubahan struktur antara mod regangan getaran trigonal bipiramid TeO_4 dan trigonal bipiramid TeO_3 dengan mod getaran membengkok pada sambungan Te-O-Te melalui pembentukan atom oksigen yang tidak bersambung. Perubahan ketara terhadap struktur adalah terbukti disebabkan oleh ko-pondop. Spektra penyerapan UV-Vis-NIR menunjukkan lapan puncak serapan yang jelas berpusat pada 470, 948, 1086, 1238, 1386, 1492, 1550 dan 1588 nm mewakili peralihan daripada keadaan asas kepada keadaan teruja. Data penyerapan ini kemudiannya digunakan dalam pengiraan pembolehubah Judd-Ofelt Ω_2 , Ω_4 and Ω_6 . Perubahan nilai Ω_2 menunjukkan sifat kovalen semula jadi ikatan manakala faktor kualiti spektroskopi mempunyai nilai di sekitar 171-251. Spektra pancaran pada suhu bilik bagi kedua-dua kaca memperlihatkan empat peningkatan puncak sehingga kepekatan ko-pondop tertentu dan kemudiannya mengurang dan ini dapat difahami menggunakan gambar rajah aras tenaga separa. Nilai maksimum bagi keratan rentas pancaran terangsang, lebar jalur perolehan dan perolehan optik bagi peralihan ${}^4\text{G}_{5/2} \rightarrow {}^6\text{H}_{9/2}$ masing-masing ialah $23.09 \times 10^{-22} \text{ cm}^2$, $28.54 \times 10^{-28} \text{ cm}^3$ dan $12.51 \text{ cm}^2 \text{ s}^{-1}$. Terdapat hubungan antara ko-pondop $\text{Sm}^{3+}/\text{Yb}^{3+}$ dengan perubahan struktur dan sifat optik bagi kaca yang dikaji. Ciri istimewa keputusan kajian ini mencadangkan bahawa kaca yang dikaji berpotensi sebagai laser keadaan pepejal dan alat fotonik lain.

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LIST OF SYMBOLS

A	-	Radiative Probability
B	-	Magnetic Induction
c	-	Speed of Light
d_{sm}	-	Inter-Nuclear Distance Between Sm-Sm Ions
e	-	Charge of Electron
E	-	Electric Field
E_{opt}	-	Optical Band Gap
ΔE	-	Urbach Energy
f	-	Oscillator Strength
H	-	Magnetic Field
I	-	Intensity
J_{ext}	-	Current Density
K	-	Wave Vector
L	-	Length
M	-	Average Molecular Weight
m	-	Mass of Electron
n_f	-	Refractive Index
N	-	Density of the Electrons
n_2	-	Non-linear Refractive Index
N_c	-	Concentration
N_A	-	Avogadro Number
R	-	Glass Constant
R_i	-	Reflection Loss
R'	-	Refractivity
S	-	Stability Factor
S_{ed}, S_{md}	-	Electric and Magnetic Dipole Line Strengths
T'	-	Transmission

T	-	Temperature
T_c	-	Crystallization Temperature
T_g	-	Glass Transition Temperature
T_m	-	Melting Temperature
t	-	Time
$\ U^{(i)}\ ^2$	-	Reduced Matrix Elements
V_m	-	Molar Volume
W	-	Weight
Z^*	-	Effective Nuclear Charge
2θ		Diffraction angle
α	-	Absorption Co-efficient
α_m	-	Polarizability
β	-	Branching Ratio
ϵ_0	-	Permittivity of Vacuum
h	-	Plank's Constant
$\chi_{(i)}$	-	Susceptibility
ρ	-	Density of Glass
σ_{em}	-	Emission Cross-Section
Ω_i	-	Judd-Ofelt Intensity Parameters
λ	-	Wavelength
ν_d	-	Abbe Number
$ (S,L)J\rangle$	-	Electronic State of an Element Defined by its Spin, Orbital and Total Momentums
σ_p^E	-	Stimulated Emission Cross-section
$\Delta\lambda_{eff}$	-	Effective Band Width

LIST OF ABBREVIATIONS

CR	-	Cross Relaxation
CW	-	Continuous Wave
DTA		Differential Thermal Analysis
ET	-	Energy Transfer
ESA	-	Excited States Absorption
ESR	-	Electron-Spin Resonance
FTIR	-	Fourier Transform Infrared
FWHM	-	Full Width at Half Maximum
GSA	-	Ground State Absorption
IR	-	Infrared
JO	-	Judd-Ofelt
LRE	-	Lightening Rod Effect
NMR	-	Nuclear-Magnetic Resonance
NR	-	Non-Radiative
NIR		Near Infrared
OD	-	Optical Density
PL	-	Photoluminescence
PLE	-	Photoluminescence Excitation
RGB	-	Red-Green-Blue
RE	-	Rare Earth
SEFS	-	Surface Enhanced Fluorescence Spectroscopy
SERS	-	Surface Enhanced Raman Spectroscopy
SHG	-	Second Harmonic Generation
SPM	-	Scanning Probe Microscopy
SPR	-	Surface Plasmon Resonance
THG	-	Third Harmonic Generation

TL	-	Thermal Lens
UC	-	Upconversion
UTM	-	Universiti Teknologi Malaysia
UV	-	Ultraviolet
VIS	-	Visible
WDM	-	Wavelength Division Multiplexing
XRD	-	X-Ray Diffraction
RMSE	-	Root Mean Square Error

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CHAPTER 1

INTRODUCTION

1.1 Background

Glass is an optically transparent solid material which is typically hard, fragile and shows glass transition. It is prepared by cooling molten constituents fast enough, to avoid visible crystallization formation. Glasses are usually poor conductor of heat and electricity and the incorporation of certain metal oxides in glasses give them colors. Glass has been found around 3000 BC in the Middle East. It is one of the oldest as well as newest material that is man's most valuable and versatile material in everyday life. A great research interest in the science and technology of glasses has received a great attention. Progress in the development of the glasses for different scientific applications was rapid and advanced through and early 20th century, in parallel with many other technology areas. Unfortunately, until 20th century most development was made experimentally by using general ideas, to guide experiments. There was no specific theory exist to support the experimental results, which raised. Many theoretical problems raised in non-crystalline solids especially, in glasses to understand their optical, structural and thermal properties due to lack of precise experimental information and absence of any specific theory to support the experimental results. There is tremendous need to accelerate the research to fill this gap. New science and technology have dynamically given great improvement in glass manufacture and their new technological applications. The main advantages of this will be to provide the fundamental base of new optical glasses with number of applications especially, tellurite-glass base solid state lasers, optical fibers and amplifiers. Great research attention is needed for the development of new material

for solid state lasers, optical switches, third order nonlinear optical materials, optical amplifiers, light emitting diodes and up-conversion glasses (El-Mallawany, 1999).

There is variety of techniques used for manufacturing of glass material such as thermal evaporation, sputtering, chemical reaction, amorphisation, irradiation, melt quenching, sol gel, etc (Chandra, 1981; Cusack & Brewer, 1987; Elliott, 1991; Rodrigues *et al.*, 1985; Thorpe & Ticha, 2001; Zallen, 2008). Among these techniques the melt quenching technique is most important and widely used technique (Beall *et al.*, 1983). In last few decades, research on glass has been very active due to the increasing awareness of industry that from fabrication point of view, glasses are better than crystalline material because glasses are isotropic material and have the same optical properties in all orientation, while crystalline have different optical properties in different orientation. Moreover glasses can more ability to incorporate the large amount of rare earth as compare to crystalline materials due to irregular arrangement of atoms, so glasses can play important role in electronics and optical devices development (Mackenzie, 1982).

In recent years, among many lasing materials, tellurite oxide (TeO_2) have attracted a large attention due to their low melting temperature, better thermal and chemical stability, high thermal expansion, high refractive index, good infrared transmission, low phonon energy and large third order non-linear optical susceptibility compare to other oxide glasses (Cherif *et al.*, 2010) (Babu *et al.*, 2009; Nii *et al.*, 1998; Wang *et al.*, 1994). TeO_2 -based glasses are used as attractive host for RE ions due their low phonon energy and wide optical window (Lines, 1990, 1991). The bonding nature in TeO_2 is covalent and its structure consists of highly deformed octahedron due to covalent character of tellurium results in two sets of Te-O distances. Four Te coordinate having four oxygen atoms at four corners of a trigonal bipyramid exist in paratellurite, $\alpha\text{-TeO}_2$. All vertices in paratellurite are shared to form three dimensional structures, which has 140° bond angle of O-Te-O, and two 2.08 Å and 1.90 Å axial and equatorial bonds distances respectively.

In modern technology, rare earth (RE) ions are very attractive candidates as active ions in many optical materials because many fluorescence states can be chosen among the 4f electronic configurations and most of them are placed in the visible region. The excited levels of these RE ions emit in the visible region, which exhibit possibly high quantum efficiency and show different quenching mechanism. This phenomenon makes RE an attractive case for the investigation of energy transfer process. The optical and structural properties of rare earth doped glasses are host material dependant. For the investigation of these properties, TeO₂-based glasses are very appropriate candidate as they accept large concentration of RE ions, exhibit large visible to infrared region transmittance window, good chemical durability, low cutoff phonon energy ($\sim 700\text{cm}^{-1}$) and high refractive index (~ 2.0). Luminescence properties of rare earth doped tellurite glasses are reported by many researchers (Babu *et al.*, 2007; Mohan Babu *et al.*, 2011).

In principle, energy transfer process may favor particular applications, such as the operation of lasers and optical amplifiers. But it may be detrimental in case of rare earth based solid state laser sources because the interaction between the active ions efficiently contributes for enhancement of the laser threshold. In particular, the investigation of energy transfer process in glasses having frequency gap in the visible region deserves great attention because rare earth ions doped glasses may present efficient visible luminescence. Enhancement of optical properties either by energy transfer between two rare earth ions or by influence of the large local field on rare earth ions positioned near the doped elements is of great technological interest. Indeed the presence of RE improved the luminescence efficiency.

In recent years, great research interest on co-doped glasses with two rare earth (RE) ions have received great attention due to their potential applications in compact optical amplifiers and solid state laser sources (Delavaux *et al.*, 1996; Elfayoumi *et al.*, 2010; Laporta *et al.*, 1999; Nilsson *et al.*, 1994; Park *et al.*, 1996; Xu *et al.*, 2006). Many experiments on co-doped glasses have been done to discuss the phenomena of radiative and non radiative transition in RE ions and researcher succeeded in realizing energy transfer in co-doped glasses (Joshi, 1995; Lim *et al.*, 2003; Qiu *et al.*, 2000). The characteristics of the non-linear optical properties of two

kinds of rare earth dopant glasses are very important for optimization and applications. These materials exhibit high infrared transmission and good thermal and mechanical stability. Among all other RE ions Sm^{3+} and Yb^{3+} are rather special because of their potential applications for high density optical memory devices. This is because of co-doping with Sm^{3+} and Yb^{3+} leads to effective energy transfer between levels close enough in $\text{Sm}^{3+} : ^6\text{F}_{11/2}$ and $\text{Yb}^{3+} : ^2\text{F}_{5/2}$.

More recently, research on $\text{TeO}_2\text{-PbO-GeO}_2$ glasses doped with Eu^{3+} and containing other rare earth dopants and Tb^{3+} doped $\text{TeO}_2\text{-ZnO-PbO-Na}_2\text{O}$ glasses with samarium co-doping are reported (de Almeida *et al.*, 2008; O'Donnell *et al.*, 2007). However, only few experimental reports exist on luminescence with tellurite based glasses co-doped with two different rare earth elements. In these experiments, the samples are excited by ultraviolet light with frequency larger than the band gap frequency of the glass. Luminescence band from 400 to 980 nm are observed due to radiative transition associated to the RE ions. The introduction of the co-doping of two different rare earth elements in glasses has been exploited to enhance the luminescence efficiency. Linear and nonlinear optical properties of the two or more rare earth ions doped glasses may cause this enhancement. For instance, the enhancement of $\text{Er}^{3+}/\text{Yb}^{3+}$ luminescence was reported in tungsten tellurite glasses in 2006 (Bjurshagen *et al.*, 2006). Later, experiments were presented where energy transfer process between two rare earth ion change the luminescence of RE in lithium borate and zinc tellurite glasses (Elfayoumi *et al.*, 2010; Jin *et al.*, 2007), but there are not many examples of $\text{Sm}^{3+}/\text{Yb}^{3+}$ doped tellurite glasses especially sodium tellurite glasses.

Presently, this approach to increase the luminescence in RE ions co-doped glasses receiving renewed attention due to large interest in photonics. Among the technologically important glasses available, the TeO_2 -based glasses are promising candidates for the studies of luminescence enhancement by the energy transfer (ET) between two RE ion. Lately, these glasses with enhanced optical and mechanical properties have been synthesized both for passive applications, multispectral imaging and active application for rare earth doping. The goal of all these studies is to

investigate the influence of RE ions co-doped on enhancing the optical properties of glasses.

Judd-Ofelt (J-O) theory (Judd, 1962; Ofelt, 2004) is mostly used to analyze the RE ions surrounding environment when doped into a glass matrix. Many parameters such as radiative life times, branching ratios, and emission cross-section, driven from the absorption spectra, can be studied by this Judd-Ofelt theory. Interests on linear and nonlinear optical properties of glasses with two or more RE ions are ever growing to achieve improved luminescence. In this view, the present work focuses on the observation and investigation of ET between Sm^{3+} and Yb^{3+} ions and their influences on concentration dependant optical behavior, which is a new study. Moreover the theoretical values calculated by J-O theory are compared with the experimental values.

1.2 Problem Statement

Enhancing the luminescence intensity and optical gain by inhibiting the concentration quenching effects remains challenging. Since the concentration quenching in the single rare earth doped materials limits their emission cross-section, the efforts have been tuned to increase the emission cross section of RE ions by developing co-doping methods (Guo *et al.*, 2010; Silver *et al.*, 2001). In this way, energy transfer mechanism between two different rare earth ions in the host would contribute to enhance or quench the visible emissions. Although there are some reports on co-doped tellurite glasses, but there is no report about the thermal structural and optical characterization of $\text{Sm}^{3+}/\text{Yb}^{3+}$ co-doped sodium tellurite glasses. Controlled co-doping for the enhancement of luminescence and emission cross section are not well studied especially for $(79.5-x)\text{TeO}_2-20\text{Na}_2\text{O}-x\text{Sm}_2\text{O}_3-0.5\text{Yb}_2\text{O}_3$, where $x=0, 0.5, 1.0, 1.5$ and 2.0 mol %. And $(79.5-y)\text{TeO}_2-20\text{Na}_2\text{O}-0.5\text{Sm}_2\text{O}_3-y\text{Yb}_2\text{O}_3$, where $y=0, 0.5, 1.0, 1.5$ and 2.0 mol% compositions. Further research is required to understand the effect of $\text{Sm}^{3+}/\text{Yb}^{3+}$ co-doping on the possibility of enhancing the optical properties and effect of co-dopants on their structure. Despite much research on binary glasses the

mechanism for enhanced luminescence due to co-doping is far from being understood and no systematic J-O theory based calculation exists for this glass system.

Theoretical understanding using J-O theory can render deep insight into the problem. This requires careful sample preparation and thorough characterization using thermal and spectroscopic means. In order to have better theoretical understanding of the influence of $\text{Sm}^{3+}/\text{Yb}^{3+}$ ions in tellurite glasses different radiative parameters such as transition cross section, branching ratio, radiative and non-radiative decay rates and transition probabilities are needed to be evaluated from the absorption and emission data within the framework of J-O theory. These parameters will bring special knowledge in designing many optical devices such as up-converter, color display lasers and fiber amplifiers.

1.3 Objectives

1. To synthesize two series of $\text{Sm}^{3+}/\text{Yb}^{3+}$ doped sodium tellurite glasses by melt quenching technique having composition:
 - i. $(80-x)\text{TeO}_2-20\text{Na}_2\text{O}-x\text{Sm}_2\text{O}_3-0.5\text{Yb}_2\text{O}_3$, where $x=0, 0.5, 1.0, 1.5$ and 2.0 mol %.
 - ii. $(80-y)\text{TeO}_2-20\text{Na}_2\text{O}-0.5\text{Sm}_2\text{O}_3-y\text{Yb}_2\text{O}_3$, where $y=0, 0.5, 1.0, 1.5$ and 2.0 mol%.
2. To determine the amorphous nature of glasses.
3. To determine the physical thermal, structural and optical parameters due to co-doping.
4. To determine the effect of co-doping on physical, thermal, structural and optical properties.
5. To calculate the intensity and radiative parameters using Judd-Ofelt theory.

1.4 Scope of Study

The scope of present study includes

1. The melt quenching technique is used to prepare the $\text{Sm}^{3+}/\text{Yb}^{3+}$ co-doped sodium tellurite glasses with different composition to optimize the co-doping.
2. Amorphous nature of glasses is examined by X-ray diffraction (XRD) technique.
3. Density and molar volume are calculated for physical characterization.
4. Thermal characterization is accomplished in terms of melting temperature, crystallization temperature, glass transition temperature and thermal stability of these glasses by using Differential thermal analysis (DTA) technique.
5. Structural behavior in terms of vibration bands of these glasses is determined by Fourier transform infrared (FTIR) spectroscopy and Raman spectroscopy.
6. Optical absorption and emission are measured by ultraviolet visible near infrared (UV-VIS-NIR) and photoluminescence (PL) spectroscopy. .
7. Special attention is also given to the calculation of intensity and radiative parameters of these glasses by using Judd-Ofelt theory for laser applications.

1.5 Significance of Study

The present research work is important for fundamental understanding of structural, optical and thermal properties in materials science. The mechanism of the linear optical behaviors due to co-doping will be clearly understood and quantified on the base of absorption and emission analysis. Our detail research methodology about physical, thermal, structural and optical characterization along with intensity and radiative parameters calculation can make accurate qualitative as well as quantitative predictions regarding the unusual and enhanced optical properties in tellurite glasses. Information generated through these studies will be highly useful to science and/or community. Moreover, controlled and optimized $\text{Sm}^{3+}/\text{Yb}^{3+}$ co-doping in the present glass system for enhanced optical properties can nominate this

glass system for wide range of applications in solid state lasers and many other photonics devices.

1.6 Thesis Organization

This thesis describes the preparation and characterization of Sm^{3+} doped sodium tellurite glasses co-doped with Yb^{3+} . Most simple and yet accurate conventional melt quenching technique is used to prepare the glass samples. The thesis consists of five chapters. Chapter one presents a brief introduction and background of the research with its topical importance. The objectives, scope of studies, problem statement and significances along with the choice of the glass material are provided in this chapter. Chapter two describes important theories and review of relevant literatures. The detail methodologies are dealt in Chapter three where glass preparation technique and characterization methods are presented. Main results, analyses, discussions and comparison with existing works are summarized in Chapter four. Special attention is focused on the effect of $\text{Sm}^{3+}/\text{Yb}^{3+}$ co-doping on the optical and structural properties of sodium tellurite glasses. Finally, Chapter five renders conclusion based on the results and facilely discusses further outlook. The calculation of some physical properties, description of instruments used in the research and list of publications are appended in the Appendix.

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