

TITANIUM DIOXIDE NANOPARTICLES MEDIATED SURFACE
PLASMON RESONANCE ASSISTED OPTICAL PROPERTIES OF ERBIUM-
DOPED TELLURITE GLASS

NUR NABIHAH BINTI YUSOF

UNIVERSITI TEKNOLOGI MALAYSIA

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DOPED TELLURITE GLASS

NUR NABIHAH BINTI YUSOF

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ABSTRACT

Low emission and absorption cross-section of rare earth (RE) ion doped tellurite glasses that limit them from making solid state lasers needs further improvement. Combination of metal nanoparticles (NPs) with RE ions in the glass is proven to be prospective for overcoming such limitations. Via tuneable surface plasmon resonance (SPR) of metal NPs, an alteration in the optical properties of such glasses is feasible. In this view, present thesis takes an attempt to determine the mechanism of metal NPs SPR mediated modification in the optical properties of Erbium (Er^{3+}) ions by embedding Titanium dioxide (TiO_2) NPs into the tellurite glass. Five glass samples are prepared using melt-quenching technique with the composition $(69-x)\text{TeO}_2-20\text{ZnO}-10\text{Na}_2\text{O}-1\text{Er}_2\text{O}_3-x\text{TiO}_2$, where $x = 0.1, 0.2, 0.3, 0.4$ mol%. Prepared samples are thoroughly characterized using X-ray diffraction (XRD), transmission electron microscope (TEM), energy dispersive X-ray (EDX), Fourier transform infrared (FTIR), Raman, ultraviolet-visible-near infrared (UV-Vis-NIR) and photoluminescence (PL) measurements. XRD pattern of achieved transparent glass samples confirmed their amorphous nature and TEM images revealed the existence of TiO_2 NPs with average size between 16 to 22 nm. EDX spectra displayed the presence of Ti element in the sample together with other appropriate elemental traces. FTIR and Raman spectra showed different vibration modes of Te-O bond (bending, asymmetric and symmetric stretching) in the structural unit of TeO_4 and TeO_3 . Glass containing 0.4 mol% of TiO_2 NPs displayed an enhancement in the Raman signal by a factor of 1.56 to 3.58 for the bands centered at 388, 495, 673, 758 and 845 cm^{-1} . This intensity enhancement is mainly attributed to the TiO_2 NPs SPR assisted effects. Meanwhile, UV-Vis-NIR spectra exhibited ten absorption bands centred at 407, 444, 452, 489, 522, 552, 653, 800, 976 and 1532 nm. Absorption spectra of TiO_2 NPs manifested two plasmon bands at 552 (transverse mode) and 580 nm (longitudinal mode). PL spectra demonstrated three prominent bands centred at 525, 545 and 660 nm, which are allocated to the Er^{3+} ion transition from the excited levels ($^2\text{H}_{11/2}$, $^4\text{S}_{3/2}$ and $^4\text{F}_{9/2}$) to the ground level ($^4\text{I}_{15/2}$). Furthermore, glass containing 0.2 mol% of TiO_2 NPs revealed PL intensity enhancement by a factor of 30.00, 28.57 (for green bands) and 19.60 (for red band). This observation is ascribed to the SPR effect of TiO_2 NPs which created strong local electric field in the vicinity of Er^{3+} ion. Green emission showed the highest enhancement due to its appearance in the immediate proximity of plasmon band. It is asserted that the incorporation of TiO_2 NPs in the glass produced plasmonic effect and thereby altered the UV-Vis-NIR absorbance and improved the emission intensity of the samples. A correlation between SPR effect and surface enhance Raman scattering (SERS) is established. Present functional glass composition may be useful for device applications especially in photovoltaic and solid state laser.

ABSTRAK

Keratan rentas penyerapan dan pancaran yang rendah bagi kaca tellurite didop dengan ion nadir bumi (RE) telah membataskannya daripada menjadi laser keadaan pepejal dan memerlukan penambahbaikan seterusnya. Gabungan zarah nano logam (NPs) dengan ion RE dalam kaca telah terbukti mampu menangani keterbatasan masalah ini. Melalui resonans plasmon permukaan (SPR) boleh tala bagi logam NPs, pengubahsuaian sifat optik kaca tersebut barangkali mampu dilaksanakan. Berdasarkan pandangan ini, tesis ini cuba untuk menentukan mekanisma SPR logam NPs yang telah menjadi pengantaraan pengubahsuaian terhadap perubahan sifat optik dopan Erbium (Er^{3+}) dengan mencampurkan zarah nano Titanium dioksida (TiO_2) ke dalam kaca tellurite. Lima sampel kaca telah disediakan menggunakan teknik perlindungan leburan dengan komposisi $(69-x)\text{TeO}_2-20\text{ZnO}-10\text{Na}_2\text{O}-1\text{Er}_2\text{O}_3-x\text{TiO}_2$, dengan $x = 0.1, 0.2, 0.3, 0.4$ % mol. Sampel yang disediakan telah menjalani pencirian menggunakan pengukuran pembelauan sinar-X (XRD), mikroskop elektron transmisi (TEM), serakan tenaga sinar-X (EDX), inframerah transformasi Fourier (FTIR), Raman, ultra ungu-cahaya nampak-inframerah dekat (UV-Vis-NIR) dan kefotopendarcahayaan (PL). Corak XRD bagi sampel kaca lutsinar yang dihasil mengesahkan ia adalah bersifat amorfus dan imej TEM pula telah mendedahkan kewujudan NPs TiO_2 dengan saiz purata antara 16 hingga 22 nm. Spektrum EDX memaparkan kehadiran unsur Ti dalam sampel bersama unsur lain yang berpadanan. Spektrum FTIR dan Raman menunjukkan mod getaran berbeza bagi ikatan Te-O (pelenturan, regangan tidak simetri dan simetri) pada struktur unit TeO_4 dan TeO_3 . Kaca yang mengandungi 0.4 % mol NPs TiO_2 memaparkan peningkatan isyarat Raman sebanyak 1.56 hingga 3.58 kali ganda bagi jalur berpusat disekitar 388, 495, 673, 758 dan 845 cm^{-1} . Peningkatan isyarat ini adalah disebabkan kesan bantuan SPR NPs TiO_2 . Manakala, spektrum UV-Vis-NIR mempamerkan sepuluh jalur penyerapan berpusat di 407, 444, 452, 489, 522, 552, 653, 800, 976 dan 1532 nm. Spektrum penyerapan NPs TiO_2 adalah manifestasi dua jalur plasmon pada 552 (mod melintang) dan 580 (mod membujur). Spektrum PL mempamerkan tiga jalur utama berpusat pada 525, 545 dan 660 nm berpadanan dengan peralihan ion Er^{3+} dari keadaan teruja ($^2\text{H}_{11/2}$, $^4\text{S}_{3/2}$ dan $^4\text{F}_{9/2}$) ke keadaan dasar ($^4\text{I}_{15/2}$). Malahan, kaca yang mengandungi 0.2 % mol NPs TiO_2 mendedahkan peningkatan keamatan PL sebanyak 30.00, 28.57 kali ganda (bagi jalur hijau) dan 19.60 kali ganda (bagi jalur merah). Cerapan ini berpunca daripada kesan SPR dari NPs TiO_2 yang menghasilkan medan elektrik setempat yang kuat disekitar ion Er^{3+} . Pancaran cahaya hijau menunjukkan peningkatan tertinggi kerana kejadiannya berhampiran dengan jalur plasmon. Perlu ditegaskan bahawa kemasukan NPs TiO_2 ke dalam kaca menghasilkan kesan plasma yang mengubah penyerapan UV-Vis-NIR dan meningkatkan keamatan pancaran sampel. Hubungkait antara SPR dan peningkatan permukaan penyerakan Raman (SERS) telah dikenalpasti. Fungsi komposisi kaca semasa dalam kajian ini berkemungkinan berguna untuk aplikasi peralatan terutamanya dalam fotovoltla dan laser keadaan pepejal.

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LIST OF ABBREVIATIONS

Au	-	Gold
Ag	-	Silver
BO	-	Bridging oxygen
CCD	-	Charge-coupled device
Ce	-	Cerium
CXA	-	Computer-assisted X-ray analyzer
CCD	-	Charge-coupled device
DC	-	Down-Conversion
Dy	-	Dysprosium
Er ³⁺	-	Erbium
EDX	-	Energy dispersive X-ray
ET	-	Energy transfer
Eu	-	Europium
ESA	-	Excited state absorption
ETU	-	Energy transfer up-conversion
eV		Electron volt
FTIR	-	Fourier transform infrared
FET	-	Field effect transistor

FFT	-	Fast Fourier transform
Gd	-	Gadolinium
GSA	-	Ground-state absorption
HRTEM	-	High resolution transmission electron microscopy
HST	-	Hypersensitive transition
Ho	-	Holmium
IR	-	Infrared
IUPAC	-	International union of pure and applied chemistry
KBr	-	Potassium bromide
LFE	-	Local field effect
La	-	Lanthanum
Lu	-	Lutetium
LPE	-	Lone pair electron
LSP	-	Localised surface plasmon
LSPR	-	Localised surface plasmon resonance
MRP	-	Multiphonon
MEF	-	Metal enhanced fluorescence
MCA	-	Multichannel analyzer
Na	-	Sodium
NPs	-	Nanoparticles
NIR	-	Near-Infrared
NBO	-	Non-bridging oxygen
Nd	-	Neodymium
NR	-	Non-radiative
Nd-YAG	-	Neodymium-doped yttrium aluminum garnet
OH-		Hydroxyl group
Pt	-	Platinum
PL	-	Photoluminescence
Pr	-	Praseodymium

Pm	-	Promethium
PA	-	Photon avalanche
RE	-	Rare earth
R	-	Radiative decay
SEM	-	Scanning electron microscope
SPR	-	Surface plasmon resonance
SERS	-	Surface enhance Raman scattering
Sc	-	Scandium
Sm	-	Samarium
SAXS	-	Small-angle X-ray scattering spectroscopy
Te	-	Tellurite
TiO ₂	-	Titanium dioxide
TEM	-	Transmission electron microscope
Tbp	-	Trigonal bipyramid
Tp	-	Trigonal pyramid
Tb	-	Terbium
Tm	-	Thulium
UTM	-	Universiti Teknologi Malaysia
UV-Vis-NIR	-	Ultra violet-visible-near infrared
UC	-	Up-conversion
Vis	-	Visible
Xe	-	Xenon
XRD	-	X-ray diffraction
XRF	-	Fluorescence
XPS	-	X-Ray photoelectron spectroscopy
Y	-	Yttrium
Yb	-	Ytterbium
Zn	-	Zinc

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Glass represent one of the most important inorganic material in infrared photonic system due to its high-transparency and superior optical properties (Sun, 2013). Recently, RE-doped glasses attract much attention due to their high emission efficiencies as luminescence material in which the RE ions are capable to emits intense radiations in visible, IR and NIR region using suitable excitation wavelength (Ravi *et al.*, 2014). These beneficial features are useful for optical devices application such as infrared lasers and fibre amplifiers (Nogami *et al.*, 2000).

First solid-state lasers: Nd-doped glass was demonstrated in 1961 (Righini and Ferrari, 2005) and found to be efficient candidates for photonic devices such as fibre and microchip lasers, include planar waveguide as well (Raju *et al.*, 2013). Since then, glass doped with other REs like Er^{3+} receive great attention especially when transition wavelength of Er^{3+} ion at 1500nm is close to operational wavelength of optical communication. Remarkable result has been achieved by intense research on RE-doped glass to develop efficient laser host for photonic and optical devices (Righini and Ferrari, 2005).

Interest in Er^{3+} -doped glass has grown due to its potential in providing the up-converted visible fluorescence for various optical applications. Thus, enhancement of fluorescence in the visible region is an important issue. Tellurite glass has been labeled as an excellent host for Er^{3+} ions that has a low phonon frequency cut-off compared to silica glass (Kishi and Tanabe, 2006; Peng *et al.*, 2014). It suppresses non-radiative loss and subsequently increases the luminescence intensity of Er^{3+} ions in the glass (Sidebottom *et al.*, 1997). In fact, tellurite glass has non-hygroscopic properties which limit the usage of other glasses such as phosphate and borate glasses (Sidek *et al.*, 2009). However, high doping of Er^{3+} ions can cause luminescence quenching in tellurite glass. This phenomenon is attributed to the interaction of Er^{3+} ions with hydroxyl (OH^-) groups (Dai *et al.*, 2006). Accordingly, these shortcomings need to be overcome to enhance the optical response of the glass system.

Presently, vitreous materials with a combination of RE ions and metallic NPs become a fruitful method to overcome these limitations. Interestingly, the presence of metal NPs in a glass matrix is capable of intensifying the luminescence intensity and enhancing nonlinear optical properties of the material. The SPR effect is proven responsible for such significant enhancement of the optical properties of the material (de Almeida *et al.*, 2008). Au and Ag NPs are common plasmonic materials used as secondary doping materials in glass to increase luminescence properties of the host glass based on their SPR effect. (Eichelbaum *et al.*, 2008; Eichelbaum and Rademann, 2009). In addition, they are really stable and hardly oxidized compared to other metal NPs. However, due to the wide range of parameters for separate applications, it is impossible to address all the requirements using noble metals. Therefore, alternative materials are needed to optimize the performance (Guler *et al.*, 2015). Literature revealed that titanium dioxide (TiO_2) NPs also exhibit SPR properties (Abdulhalim *et al.*, 2008). However, no literature reported the incorporation of pure TiO_2 NPs as sensitizers in RE ions-doped tellurite glass. Other than that, TiO_2 NPs are also considered as abundant, low-cost materials with high chemical stability and photocatalytic activity that are safe towards both humans and the environment (Gupta and Tripathi, 2011).

Enhancement of Raman signal to several orders of magnitude due to molecule polarization is known as Surface Enhance Raman Scattering (SERS). Previous research have establish correlations between SPR and SERS enhancement by tuning SPR band using different NPs size and structure (Fleger and Rosenbluh, 2009). SPR is thought responsible for localised field enhancement to stimulate SERS (Darvill *et al.*, 2013). Of late, the SPR effects and SERS of titanium dioxide NPs (TiO₂ NPs) embedded erbium-tellurite glasses have not been explored. In addition, there is an insufficient systematic experimental and specific study particularly on SERS to explain the role of TiO₂ NPs embedded in Er³⁺-doped tellurite glasses. Therefore, the present study focus on determine the role of TiO₂ NPs mediated SPR assisted electromagnetic interaction which is responsible in enhancing the optical properties of tellurite glasses containing low-concentration of Er³⁺ ions. Furthermore, the mechanism of SPR and SERS will be addressed in accordance for their potential in nanophotonics device applications.

1.2 Problem Statement

Emission of erbium ion in tellurite glass requires high intensity and gain enhancement for diverse applications. Embedment of metallic NPs in erbium doped tellurite glass is believed able to enhance the optical response via SPR effects (Reza Dousti *et al.*, 2013a). Unlike Au and Ag NPs, a detail studies on the mechanism of SPR enhanced optical properties mediated by pure TiO₂ NPs with varying concentration in tellurite-based glass materials is not fully understood (Awang *et al.*, 2013; Reza Dousti *et al.*, 2013). Glass composition optimization due to the embedment of NPs in the glass matrix is pre-requisite to determine the modified properties. Therefore, optimization of TiO₂ NPs concentration inside erbium-doped tellurite glass need to be carried out.

Size, shape and distribution of metal NPs inside the glass significantly change the optical properties of the glass (Rivera *et al.*, 2011). The variations of metal NPs size and shape in the glass matrix containing Er^{3+} ions able to modified the luminescence properties of Er^{3+} ions by altering its local symmetries through amplification of electric field intensity and efficient energy transfer from metal NPs to Er^{3+} ions (Awang *et al.*, 2014). However no further investigations has been done to overlook the effect of changing TiO_2 NPs size and shape inside the glass containing Er^{3+} ions on the enhancement of local electric field and luminescence properties. Thus, further investigate is needed to identify the effect of different TiO_2 NPs size and shape on the optical properties of the glass in term of SPR effect.

There has been correlation between SERS and SPR effect of metal NPs with its different shape and size inside erbium-doped tellurite glass (Awang *et al.*, 2013; Reza Dousti *et al.*, 2013). Yet the relationship between the SPR effects and SERS of TiO_2 NPs in erbium-doped tellurite glasses has not been further studied. In addition, evolution of structural and optical properties in the presence of TiO_2 NPs and the role of SPR effects on overall glass properties have not reported yet. Therefore, the present study will provide new information regarding the structural and optical properties of tellurite glass doped at lower concentration of Er^{3+} ions embedded with varying concentrations of TiO_2 NPs. Correlation between SERS and SPR effect with presence TiO_2 NPs inside the Er^{3+} -doped tellurite glass will be establish. Overall, this study will provide the fundamental underlying physics of SPR in tellurite glass which useful for widespread applications.

1.3 Objectives

The objectives of this study are as follows:

- i. To synthesize a series of glass by melt quenching technique with composition $(69-x)\text{TeO}_2-20\text{ZnO}-10\text{Na}_2\text{O}-1\text{Er}_2\text{O}_3-(x)\text{TiO}_2$, where $x = 0, 0.1, 0.2, 0.3$ and 0.4 mol%.
- ii. To identify the existence of TiO_2 NPs and distinguish for different sizes and shapes in glass matrix using transmission electron microscopic (TEM) and energy dispersive X-ray (EDX) measurements.
- iii. To determine the effect of different TiO_2 NPs concentration on structural properties of the glass include SERS effect using Infrared spectroscopy and Raman spectroscopy.
- iv. To determine the role of TiO_2 NPs on absorption and emission properties of glass using ultra-violet-visible-near infrared (UV-Vis-NIR) spectroscopy and photoluminescence (PL) spectroscopy.

1.4 Scope of Study

Series of glass with composition of $(69-x)\text{TeO}_2-20\text{ZnO}-10\text{Na}_2\text{O}-1\text{Er}_2\text{O}_3-(x)\text{TiO}_2$ are prepared by melt-quenching technique. The composition of $70\text{TeO}_2-20\text{ZnO}-10\text{Na}_2\text{O}$ has been identified as promising Er^{3+} host matrix for photonic application (Souza *et al.*, 2002). Therefore, this glass composition is chosen due to its optimal performance. Tellurite has remarkable characteristic as host glass. Furthermore, ZnO will be added as modifier for better glass forming ability and reduce crystallization rates of tellurite network (Said Mahraz *et al.*, 2013). The addition of

Na₂O increase the solubility of RE ions in glass (Baki *et al.*, 2014). Metal oxide is chosen due to its chemical and mechanical stabilities (Clara Gonçalves *et al.*, 2002). Awang *et al.*, (2013) proposed that using 1.0 mol% of Er₂O₃ show the most intense UC luminescence for all spectral range in tellurite-zinc-sodium based glass system. Further addition of Er₂O₃ cause quenching effect on UC emission. Thus, 1.0 mol% concentration of Er₂O₃ is used in this glass system to avoid fluorescence quenching and to obtain optimum luminescence spectra. Accordingly, TiO₂ NPs with different concentration are incorporate into glass system to examine the SPR effect on the optical properties of the glass especially.

The amorphous nature of glass is determined using XRD. TEM is used to obtain the micrograph of NPs distribute in glass matrix and EDX analysis are performed to affirm the existence of TiO₂ NPs by tracing Ti element inside the sample. Vibrational structure of glass are determined using FTIR and Raman spectroscopy. The enhancement in Raman signal is determined to verify the correlation between SERS and SPR effect in tailoring the structural properties of glass. Meanwhile the absorption and emission spectra of glass are analyzed using UV-Vis-NIR and PL spectroscopy.

1.5 Research Significance

The relevance of present study leads to the improvement in fundamental understanding regarding mechanism of enhanced nonlinear optical properties of glass material. The systematic and controlled synthesis method is useful for large scale production of glasses at industrial level. The utilization of TiO₂ NPs instead of Au and Ag NPs is probable to extend a new knowledge regarding the effect of SPR and its relation with SERS on modification in optical properties of Er³⁺-doped ZnO-Na₂O

tellurite glasses. This beneficial knowledge contributes to the fabrication of functional materials for various applications in the field of nanophotonics, photovoltaics and biology in general meanwhile plasmonic solar cells, lasers and self-cleaning glasses, in particular. The present study is also useful to bring new insight of the plasmonic nanoglass research with desired properties for industrial application in Malaysia.

1.6 Thesis Outline

This thesis consists of five chapters and describes the modification of optical properties in Er^{3+} -doped $\text{ZnO-Na}_2\text{O}$ tellurite glass in the presence of TiO_2 NPs mediated the SPR effect. This study offers a systematic way of preparing glass using melt-quenching technique with selected glass composition and subsequent thorough characterizations in term of physical, structural and optical properties.

Chapter 1 provides brief introduction and motivation of this research. Problem statement and objective are included to show the research gap and ways to tackle such problem. The scope and significance of this research are also written in this chapter.

Chapter 2 offers general information regarding structure of tellurite glass including the role of Er^{3+} and TiO_2 NPs in glass network. The mechanism associated with SPR and SERS effect from TiO_2 NPs mediated modification in optical properties of the glass are also discussed. Theory relate to this work are included. Working principle and basic concept of instrument used in this work are presented and some overview of previous studies also attached.

Chapter 3 underscores the methodology of sample preparation and procedure executed to obtain results from instrument used. In addition, the in depth information regarding characterization in terms of physical properties, structural and optical properties of samples are emphasized.

Chapter 4 presents all the results of different characterizations include the discussion on the data analyses towards the fulfilment of the proposed objectives. The mechanisms responsible for enhancement or quenching of the sample are explained. The correlation between NPs size and spectroscopic modification are established.

Chapter 5 concludes the entire research to ensure research objectives are fulfilled. Many aspects has not fully explored due time restriction. However, the information gather from this research may beneficial for future directions.

Certain important calculations involving the methodology and the results that are not included in Chapter 4 are attached in the appendix.

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