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Preface

Spectroscopic investigations of crystals and glasses doped with lanthanide (Ln) ions play an important role in science and technology especially in laser physics. When compared to crystalline materials, non-crystalline materials (glasses) are favourable hosts for rare earth ions due to their broad inhomogeneous band widths, possible tuning to different wavelengths, high rare earth ion solubility and easy to mould to different shapes and sizes. Since Maiman observed laser from the ruby in 1960, a large number of laser materials have been developed so far. Much work has been devoted to high-energy and high-average-power lasers for many applications, such as laser ignition of Inertia Confined Fusion (ICF), material processing and various scientific research activities. Especially, the patronages offered by high-energy neodymium (Nd) doped laser system with an output wavelength at around 1060 nm ignited ICF experiments and has increased the hope for producing nuclear fusion energy to meet the world's future energy demand.

Quite recently rare earth doped glasses have attracted much attention because of their wide range applications in the areas such as up-converters, optical amplifiers and storage devices. The lasers beams produced by rare earth doped glasses are used in countless applications including surgical instrumentation, industrial materials processing, precision interferometry, meteorological monitoring, ultra-fast imaging, and fundamental research. Rare earth ions also play a critical role in energy-efficient luminescent materials such as phosphors for fluorescent lamps, cathode ray tubes (CRT's), and plasma displays. Rare earths are also used in the erbium-doped fiber amplifiers that are the essential component of worldwide telecommunications network. Rare-earth-activated electroluminescent materials may also be used to improve electro-optical applications that rely on the direct generation of either narrow or broad spectral emission from direct electrical pumping. Many computing and communication systems require real-time, wide-bandwidth information storage and signal processing solutions that can be achieved using spatial spectral holography in rare-earth-activated materials.

The number of applications for rare-earth activated optical materials in various fields makes me to study the **"Luminescent Characterization of certain Rare Earth ions (Pr^{3+}, Nd^{3+}, Ho^{3+} and Tm^{3+}) doped Lead Tungsten Tellurite glasses"** because glasses acts as best hosts for these rare earths. Among all the conventional glass systems, Tellurite glasses have their own importance because of their special properties such as low melting point, high optical transparency, high chemical stability, good rare earth ion solubility and low phonon energy (780 cm^{-1}). They are also found to be robust and moderate expensive. Tellurium based glasses with slow crystallization rate, owing to good transparency in a wide spectral region from visible to NIR region. Relatively good mechanical strength, chemical stability and high refractive indices make them as the best host materials for obtaining efficient luminescence from the doped trivalent rare-earth ions. Tungsten oxide (WO_3) is a precise noble semi conducting and transition-metal oxide which has fascinated considerable attention for several years. It is also one of the most examined and used material for electro-chromic and photo-chromic devices in which coloration and bleaching can be reversibly obtained by an electro-chemical process and has extensive applications in smart windows, display devices and sensors. Tungsten ions are also capable to influence the luminescence characteristics of rare earth ions in tellurite glasses, for the simple reason that these ions can exist in different valence states i.e., W^{6+}, W^{5+} and W^{4+} irrespective of their starting oxidation state in glasses. It is well known that heavy metal fluoride compounds such as PbF_2 having very less phonon energies (~340 cm^{-1}) when added to a host glass can reduce phonon energies of the host glass drastically.

Moreover fluoride compounds added to a glass can react and remove the OH content present in glass and helps in reducing the phonon energies of the host glass further. Considering the aforementioned scientific patronages offered by PbF_2, WO_3 and TeO_2, we prepared Lead Tungsten Telluride (LTT) glasses using these chemicals as constituent elements to study the luminescence properties of these glasses doped with different rare earth ions such as Pr^{3+}, Nd^{3+}, Ho^{3+} and Tm^{3+} at relatively large concentrations.

Motivation and Objectives of the work

The synopsis of the present thesis entitled **"Luminescent Characterization of certain Rare Earth ions (Pr^{3+}, Nd^{3+}, Ho^{3+} and Tm^{3+}) doped Lead Tungsten Tellurite glasses"** reports the preparation, characterization of the LTT glasses by using absorption and photoluminescence and lifetime spectral studies in different regions of the electromagnetic spectrum covering visible and Near Infrared regions. In general a host glass with low phonon energies can give high quantum efficiency by suppressing non-radiative decay transition rates. Therefore the ultimate aim of the present work is to prepare the tellurite based glassy systems with low phonon energies with good transparency, high thermal stability, good rare earth ion solubility and better suited for the luminescent applications.

The most important key objectives of the thesis are as follows

1. To prepare the good optical glasses doped with certain rare earth ions such as Pr^{3+}, Nd^{3+}, Ho^{3+} and Tm^{3+} for wide range of photonic device applications.
2. To study the physical properties of glasses such as refractive index, density, average molecular weight, ion concentration, mean atomic volume etc.
3. To study the structural effects of these glasses through XRD and FT-IR spectral analysis.
4. To study the photoluminescence and spectroscopic properties of these glasses through absorption, excitation, luminescence and decay spectra.
5. To evaluate radiative lifetimes, branching ratio, emission cross sections and quantum efficiencies of the prepared glasses by coupling the absorption and luminescence along with emission decay measurements.
6. To Optimize the concentration of the rare earth ion in the prepared glassy systems for visible and NIR luminescence applications.

The thesis is organized in seven chapters as summarized below:

Chapter 1:- General Introduction

This chapter briefly explain an overview on the science and technology of the glasses and spectroscopy of Ln^{3+} doped glasses along with the essential theoretical models used to investigate the absorption and photoluminescence spectra of the rare earth ions doped glasses. Utilization of Judd-Ofelt (J-O) theory needed to evaluate the radiative transition probabilities (A_R), branching ratios (β_R), radiative lifetimes (τ_R) of the fluorescent transitions of rare earth doped materials explained precisely. The correlation of photoluminescence spectral data with absorption data needed to evaluate the stimulated emission cross-sections (σ_{se}) and quantum efficiencies (η) of various excited levels has been summarized. The evaluation of CIE chromaticity colour coordinates (x, y) from the photoluminescence spectra to test the visible light tunability has also been explained.

Chapter 2:- Experimental Techniques

This chapter highlights the experimental techniques used to prepare and characterize the LTT glasses doped with different concentrations of Pr^{3+}, Nd^{3+}, Ho^{3+} and Tm^{3+} ions. Experimental measurement of physical properties such as refractive indices and densities needed to evaluate various other physical properties for the doped LTT glasses have been explained clearly. This chapter also explains the experimental techniques used to characterize the doped LTT glasses such as X-ray diffraction (XRD), Fourier transform infrared spectra (FT-IR), absorption, photoluminescence and lifetime spectra.

Chapter 3: Pr^{3+} doped Lead Tungsten Tellurite Glasses for Visible Red Lasers.

In this chapter we have discussed the preparation of LTT glasses doped with Pr^{3+} (0.01, 0.1, 0.5, 1.0 and 1.5 mol %) ions using the conventional melt quenching technique. Luminescence performance of the LTT glasses doped with Pr^{3+} ions were investigated through optical absorption and photoluminescence spectral studies. From the measured intensities of various absorption bands of these glasses, the three phenomenological J-O intensity parameters (Ω_2, Ω_4 and Ω_6) have been evaluated by using standard as well as modified J-O theory. The J-O parameters measured from modified J-O theory were used to characterize the absorption and luminescence spectra of these glasses. From this theory, various radiative properties like radiative transition probability (A_R), total transition probability (A_T), branching ratio (β_R) and radiative lifetime (τ_R) have been evaluated for the fluorescent levels of Pr^{3+} in these glasses. The emission spectra show five emission bands in visible region for which the effective band widths ($\Delta\lambda_P$) and emission cross-sections (σ_{se}) have been evaluated. Among all the five emission transitions, a transition ${}^3P_0 \rightarrow {}^3F_2$ is more intense and falling in red region. The visible emission spectra, stimulated emission cross-sections and branching ratios observed for all these glasses suggest the feasibility of using these glasses as lasers in red region. The CIE chromaticity co-ordinates were also evaluated from the emission spectra to understand the suitability of these materials for red emission. From the absorption, emission and CIE chromaticity measurements, it was found that 1mol% of Pr^{3+} ion concentration is quite suitable for LTT glasses to develop bright red lasers from these glasses.

These results described in this chapter were published in an international Journal "Ceramics International 40(2014)6261–6269".

Chapter 4:- Spectroscopic studies of Nd^{3+} doped Lead Tungsten Tellurite Glasses for the NIR emission at 1062 nm

In this chapter, we explained in detailed the preparation and characterization of LTT glasses doped with different concentrations of Nd^{3+} ions (0.1, 0.5, 1.0 and 1.5 mol %). The above glasses were prepared by using the melt quenching technique to study the absorption, emission and decay spectral profiles with an aim to understand the lasing potentialities of these glasses. From the absorption spectra, the J-O parameters Ω_λ (λ=2, 4 and 6) are evaluated and in turn used to calculate the transition probability (A_R), total transition probability (A_T), radiative lifetime (τ_R) and branching ratios (β_R) for prominent emission levels of Nd^{3+}. The emission spectra recorded for LTT glasses gives three emission transitions ${}^4F_{3/2}\rightarrow{}^4I_{9/2}$, ${}^4F_{3/2}\rightarrow{}^4I_{11/2}$ and ${}^4F_{3/2}\rightarrow{}^4I_{13/2}$ for which effective band widths ($\Delta\lambda_P$) and stimulated emission cross-sections (σ_{se}) are evaluated. Branching ratios (β_R) measured for all the LTT glasses show that ${}^4F_{3/2}\rightarrow{}^4I_{11/2}$ transition is quite suitable for lasing applications. The intensity of emission spectra increases with increase in the concentrations of Nd^{3+} up to 1.0 mol% and beyond concentration quenching is observed. Relatively higher emission cross-sections and branching ratios observed for the present LTT glasses over the reported glasses suggests the feasibility of using LTT glasses for infrared laser applications. From the absorption, emission and decay spectral measurements, it was found that 1.0 mol% of Nd^{3+} ion concentration is aptly suitable for LTT glasses to give a strong NIR laser emission at 1062 nm. These results are also compared with the reported Nd^{3+} doped glass systems along with Nd^{3+} doped commercial laser glass LG-750.

The interesting results explained in this chapter were published in an International journal "Optical Materials 39(2015) 8-15".

Chapter 5:- Holmium doped Lead Tungsten Tellurite Glasses for Green Luminescent Applications

In this chapter, LTT glasses doped with different concentrations of Holmium with composition (60-x) TeO_2 + 25 WO_3 + 15 PbF_2 + x Ho_2O_3 (x=0.1, 0.5, 1.0, 1.5, 2.0, 2.5 mol %) were prepared by conventional melt quenching technique and characterized to understand their visible emission characteristic futures using optical absorption and photoluminescence spectral studies. The J-O parameters Ω_λ (λ=2, 4 and 6) measured from the absorption spectral features were used to evaluate radiative properties such as transition probability (A_R), branching ratio (β_R) and radiative lifetimes (τ_R) for the prominent fluorescent levels of Ho^{3+} in LTT glasses. The photoluminescence spectra recorded for all the Ho^{3+} doped LTT glasses at an excitation wavelength 452 nm gives three prominent emission transitions $^5F_4 \rightarrow {}^5I_8$, $^5F_5 \rightarrow {}^5I_8$ and $^5F_4 \rightarrow {}^5I_7$, of which $^5F_4 \rightarrow {}^5I_8$ observed in visible green region (546 nm) is relatively more intense than the other two transitions. The intensity of $^5F_4 \rightarrow {}^5I_8$ emission transition in these glasses increases up to 1mol% of Ho^{3+} ions and beyond concentration quenching is observed. Branching ratios (β_R) and emission cross-sections (σ_{se}) were evaluated for the intense emission transition $^5F_4 \rightarrow {}^5I_8$ in these glasses to understand the luminescence efficiency in visible green region (546 nm). The CIE chromaticity coordinates were also evaluated in order to understand the suitability of these glasses for visible luminescence. From the measured emission cross-sections and CIE coordinates, it was found that 1 mol% of Ho^{3+} ions in LTT glasses are most suitable for visible green luminescence in principle. **"The important findings of this chapter were published in an international journal "Journal of luminescence 163(2015)54-71."**

Chapter 6:- Luminescence Spectral Studies of Tm^{3+} ions doped Lead Tungsten Tellurite glasses for Visible Red and NIR applications.

In this chapter, LTT glasses doped with different concentrations of Tm^{3+} ions of composition (60-x) TeO_2 + 25 WO_3 + 15 PbF_2 + x Tm_2O_3 (x=0.1, 0.5, 1.0, 1.5, 2.0, 2.5 mol %) were synthesized through melt quenching technique and characterized by using optical absorption, photoluminescence to understand the lasing potentialities and their utility for green light emitting diodes (LEDs). J-O theory has been applied to the recorded absorption spectral profiles needed to calculate the J-O parameters Ω_λ (λ=2, 4 and 6) necessary to evaluate radiative properties such as transition probability (A_R), radiative lifetimes (τ_R), branching ratios (β_R) and quantum efficiency (η) for the prominent fluorescent levels. The emission spectra recorded for these glasses gives two emission transitions $^1G_4 \rightarrow {}^3F_4$, and $^3H_4 \rightarrow {}^3H_6$ for which effective band widths ($\Delta\lambda_p$), experimental branching ratios (β_{exp}) and stimulated emission cross-sections (σ_{se}) are evaluated. The intensities of emission peaks quenches at higher concentrations (> 0.5 mol %) and maximum intensity is observed at 0.5 mol %. Among the observed emission transitions, a transition at 650 nm ($^1G_4 \rightarrow {}^3F_4$) indicates the lasing potentialities. The CIE chromaticity co-ordinates of these glasses further suggests the near Red light generation capabilities in principle. The interesting results highlighted in this chapter were communicated to an International Journal “Journal of Luminescence” quite recently.

Chapter 7:- Conclusion

This chapter summaries the investigation of results obtained for Pr^{3+}, Nd^{3+}, Ho^{3+} and Tm^{3+} doped LTT glasses. These results were compared with the similar ions doped systems data available in the literature. The future scope of the work plan to improve the luminescence efficiencies of the LTT glasses by the optimization of co-dopant ions concentration has also been explained. Different applications of these glasses in the various fields of science and technology have been discussed.

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Symbol	**Description**
T_g	Glass transition temperature (°C)
T_m	Melting temperature (°C)
Nd^{3+}	Neodymium
Pr^{3+}	Praseodymium
Ho^{3+}	Holmium
Tm^{3+}	Thulium
Sm^{3+}	Samarium
Ω_λ (λ=2, 4 and 6)	Judd Ofelt Intensity Parameters (cm^2)
n_d	Refractive index
D	Density (g/cm^3)
V_m	Molar Volume (cm^3/mol)
$\overline{M}$	Average molecular weight (g)
N	Nd^{3+} ion concentration ($ions/cm^3$)
M_v	Mean atomic volume ($g/cm^3/atom$)
$\epsilon - 1$	Optical dielectric constant (farads/m)
ϵ	Dielectric constant (farads/m)
R	Reflection losses (%)
R_m	Molar refraction (cm^{-3})
r_p	Polaron radius (Å)
r_i	Interatomic distance (Å)
α	Molecular electronic polarizability (cm^3)
F	Field strength (cm^{-2})
NA	Numerical aperture
Λ_{th}	Optical basicity
α, β and γ	Configuration interaction parameters (cm^{-1})
B	Nephelauxetic ratio
Δ	Bonding parameter
λ	Wavelength (nm)

f_{exp}	Experimental oscillator strength
M	Mass of electron (g)
C	Velocity of light (cm/s)
N	Avagadro's Number
E	Charge of the electron
$\varepsilon(\vartheta)$	Molar absorptivity of a band
ϑ	Wave number in cm^{-1}
$S_{ED}(\Psi_J, \Psi'_{J'})$	The line strength of the electric dipole transition
$\|U^{(\lambda)}\|^2$	Doubly reduced matrix elements
L	Orbital angular momentum
S	Spin angular momentum
J	Total angular momentum
g	Lande's factor
$S_{MD}(\Psi_J, \Psi'_{J'})$	Magnetic dipole line strength
f_{cal}	Calculated oscillator strength
δ_{rms}	Root mean square deviation
χ	Spectroscopic quality factor
Ψ_J	Ground state
$\Psi'_{J'}$	Excited state
A_R	Radiative transition probability (s^{-1})
A_T	Total radiative transition probability (s^{-1})
τ_R	Radiative lifetime (s)
τ_{exp}	Experimental lifetime (s)
β_R	Radiative branching ratio
β_{exp}	Experimental branching ratio
σ_{se}	Stimulated emission cross-section (cm^2)
H	Quantum efficiency
λ_p	Transition or emission peak wavelength (nm)

$\Delta\lambda_{eff}/\Delta\lambda_{\mathrm{p}}$	Effective linewidth/ band width (nm)
W_{NR}	Non-radiative decay rate (s^{-1})
W_{MPR}	Non-radiative decay rate due to multi-phonon relaxation (s^{-1})
W_{ET}	Non-radiative decay rate due to energy transfer between donor to donor or donor to acceptor (s^{-1})
W_{CQ}	Non-radiative decay rate due to concentration quenching (s^{-1})
W_{OH}	Non-radiative decay rate due to hydroxyl (OH) group (s^{-1})
$I(t)$	Luminescence decay
T	Time after excitation (s)
τ_0	Intrinsic decay time of the donors in the absence of acceptors (s)
Q	Energy transfer parameter
S	Interaction factor
Γ	Euler's function
N_0	Acceptors concentration
R_0	Critical transfer distance (Å)
C_{DA}	Elementary energy transfer of direct donor acceptor interaction between the trivalent rare earth ions at the distance R_0 ($cm^6\,s^{-1}$)
(x, y)	Colour co-ordinates

List of Abbreviations

Abbreviation	**Description**
MALDI	Matrix Assisted Laser Desorption Ionization Imaging
HR MAS NMR	High Resolution Magic Angle Spinning Nuclear Magnetic Resonance
FT-IR	Fourier Transform Infra-Red spectroscopy
AAS	Atomic Absorption Spectroscopy
RE	Rare Earth
IR	Infra-Red
UV	Ultra Violet
VUV	vacuum ultraviolet
LED	Light Emitting Diode
ED	Electric Dipole
MD	Magnetic Dipole
J-O	Judd-Ofelt
NBO	Non Bridging Oxygens
XPS	X-ray Photoelectron Spectroscopy
UV-vis-NIR	Ultraviolet-visible-Nearinfrared
XRD	X-ray Diffraction
CIE	Commission Internationale de I‟Eclairage
LTT	Lead Tungsten Tellurite
ppm	Parts Per Million
PL	Photoluminescence
NR	Non-Radiative
MPR	Multi Phonon Relaxation

List of Photographs