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Determination of the Spectroscopic Properties and Laser Performance of Thulium-Doped Fluorophosphate Glass

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Abstract: - In this work, we investigated the spectroscopic and laser performance of thulium-doped new fluorophosphate glass. Spectroscopic parameters were determined using Judd-Ofelt theory, including absorption cross-sections, Judd-Ofelt parameters, radiative transitions probabilities, branching ratios, and radiative lifetimes. Analysis of Judd-Ofelt parameters revealed that Tm^{3+} ions form less covalent bonds with nearest neighbours compared to similar glass compositions. The spectroscopic quality factor (Ω_4/Ω_6) was found to be 2.63, suggesting strong potential for laser applications. Using McCumber's theory, we calculated the emission cross-section and subsequently determined the gain cross-section and gain coefficient for the $^3F_4 \rightarrow {}^3H_6$ laser transition. Results demonstrated positive gain when population inversion ration exceeded 0.2. Furthermore, we observed a wavelength shift in the gain coefficient with increasing population inversion ratio, characteristic of quasi-three-level laser systems. Thes findings suggest that this Tm^{3+} doped fluorophosphate glass is a promising candidate for optoelectronic applications, particularly in lasers and optical amplifiers.

Keywords: Thulium-doped glass, fluorophosphate glass, Judd-Ofelt theory, McCumber theory, spectroscopic properties, laser gain.

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I. INTRODUCTION

Solid-state materials serve as excellent hosts for lasers and optical amplification application. Among these materials, glasses doped with rare earth ions presents as good hosts [1,2]. Glass, discovered millennia ago, continues to be fundamental in modern applications, particularly in specialized electronic and optical technologies. It's versatility and properties can be enhanced through doping with rare earth ions (RE^{3+}), which provide unique optical characteristics [3].

The partially filled 4f electron configuration of rare earth ions (RE^{3+}), gives rise to energy levels that produce wavelengths ranging from ultraviolet to infrared, making them promising candidates for a diverse range of photonic applications, such as sensors, color displays, holography, upconversion lasers, infrared detection, and data storage [4-9].

Among various glass composition, fluorophosphate glasses offer important advantages. They are easily prepared by introducing selected metal halides into a polyphosphate glass. These glasses are characterized by a high content of rare earth ions, low phonon energy, transmittance from ultraviolet to infrared, and a low nonlinear refractive index [8-10].

Recent interest has focused on mid-infrared lasers emitting at 2µm, particularly on using of thulium (Tm^{3+}) and holmium (Ho^{3+}) ions, owing to their potential applications in several fields such as eye safe laser radars, remote sensing, military, atmospheric pollution detection, medical surgery [8,11-13]. Efficient 2µm emission required an appropriate rare earth ions and host materials. Thulium doped glasses are particularly promising for developing laser operating in multiple wavelength ranges: infrared (~ 1.8µm), blue (~450nm) and S-Band (1460-1530nm) [14,15].

This study aims to investigate the spectroscopic properties of thulium-doped fluorophosphate glass using absorption spectra and Judd-Ofelt theory, with particular focus on evaluation its potential for laser applications.

II. GLASS PREPARATION AND PHYSICAL MEASUREMENTS

Fluorophosphate glass (labelled NPZS) doped with 1% mol thulium was prepared using high-purity powders: $NaPO_3$, SrF_2, ZnF_2, and TmF_3. Synthesis includes a series of classical steps: mixing of starting materials, melting, fining, casting and annealing as described in [8]. The above batch composition is melted in a platinum tube under room atmosphere. Temperature of glass transition T_g and temperature of onset crystallization T_x were determined by differential scanning calorimetry (DSC) using DSC TA Instrument with a programmed heating rate of 10°C/min. Densities were measured by Archimedean method. Absorption spectrum was recorded using a double

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beam spectrophotometer UV-Vis - Near IR CARY 5G brand operating between 200 and 3000 nm with a spectral resolution of 0.1 nm. All the measurements were carried out at room temperature.

III. THEORETICAL BACKGROUND

Judd-Ofelt theory, developed by Judd [16] and Ofelt [17], is widely used method for calculating spectroscopic properties of trivalent rare earth ions. While the original articles provide detailed theory, we focus on essential formulas for determining oscillator strengths, Judd-Ofelt parameters, radiative transition probabilities, branching ratios, and radiative lifetimes.

The absorption spectra of trivalent rare earth ions show primarily electric dipole transitions within the majority of the intra-configurational *f-f* transitions, with some magnetic dipole transitions also present [8,18]. The intensities of these transitions can be quantified through their oscillator strengths (f_{mes}) for each J →J' transition. The oscillator strength is directly proportional to the band absorption intensity, providing a quantitative measure of transition strength expressed as:

$$f_{\mathrm{mes}} = \frac{\mathrm{m_e c^2}}{\mathrm{\pi e^2 N}} \int \frac{\alpha(\lambda) d\lambda}{\lambda^2} \tag{1}$$

Where: f_{mes} is the measured oscillator strength, N is the ion concentration, m_e is the electron mass, c is the celerity of the light in the vacuum, e is the electron charge, $\alpha(\lambda)$ is the absorption coefficient at wavelength λ and the integral represents the area under the absorption curve.

The absorption coefficient can be calculated using the formula:

$$\alpha = \frac{\ln 10}{x} . \mathrm{OD}(\lambda) \tag{2}$$

where OD(λ) is the optical density of the absorption spectrum.

The relation between absorption cross-sections σ_{ab} and the absorption coefficient can be expressed by:

$$\sigma_{\mathrm{a}b} = \frac{\alpha(\lambda)}{\mathrm{N}} \tag{3}$$

The calculated oscillator strength can be given by:

$$f_{cal} = \frac{8\pi^2 mc}{3h(2J+1)\lambda} \frac{(n^2+2)}{9n} S_{ed} \tag{4}$$

Where S_{ed} is the electric dipole line strength expressed as:

$$S_{ed} = e^2 \sum_{k=2,4,6} \Omega_k \left| \langle J' \| U^{(k)} \| J \rangle \right|^2 \tag{5}$$

Ω_k (k = 2, 4 and 6) are the Judd-Ofelt parameters which are dependent on both the chemical environment and the lanthanide ion [18,19]. The factor $(n^2+2)/9n$ accounts for the influence of the dielectric medium, with refractive index n, on the rare earth ion. the $U^{(k)}$ terms represent the components of the reduced tensorial operator, which are independent of ligand field. The $U^{(k)}$ values are usually considered independent of host material and can be found tabulated in literature [20]. h denotes Planck's constant and λ represents the average wavelength of the transition.

The Ω_k values are empirically determined by comparing calculated oscillator strengths (f_{cal}) with experimental values obtained from absorption spectra (f_{mes}). Using a least-squares approximation, a system of q equations (where q is the number of absorption bands) is solved to determine the values of the parameters Ω_k. The accuracy of the fit is given by the root mean square deviation (RMS):

$$\mathrm{RMS} = \sqrt{\frac{\sum(\mathrm{f_{cal}\text{-}f_{mes}})^2}{\mathrm{q\text{-}3}}} \tag{6}$$

Radiative transitions probabilities are given by:

$$A_{rad}(J,J') = \frac{64\pi^4}{3h(2J+1)\lambda^3}\left[\frac{n(n^2+2)^2}{9}\right]S_{ed} \tag{7}$$

The branching ratios can be to obtain by:

$$\beta = \frac{A_{rad}(J,J')}{\sum_{J'} A_{rad}(J,J')} \tag{8}$$

The radiative lifetime of the level *J* is given by:

$$\tau_{rad} = \frac{1}{\sum_{J'} A_{rad}(J,J')} \tag{9}$$

The emission cross-section can be calculated from the absorption cross-section, using the McCumber method, by the following relation [21]:

$$\sigma_{em} = \sigma_{ab}\frac{z_l}{z_u}\exp\left[\frac{hc}{kT}\left(\frac{1}{\lambda_{ZL}} - \frac{1}{\lambda}\right)\right] \tag{11}$$

where Z_l and Z_u are the partition functions for the lower and upper energy levels involved in the considered optical transition, respectively. T is the temperature (room temperature), k is the Boltzmann constant and λ_{ZL} is the wavelength of zero-phonon line (transition between the lowest Stark sublevels of the emitting and receiving multiplets).

Using the absorption and emission cross sections (σ_{abs} and σ_{em}), we can calculate the gain coefficient by:[22]

$$\gamma(\lambda) = N[P\sigma_{em}(\lambda) - (1-P)\sigma_{ab}(\lambda)] \tag{12}$$

where p corresponds to the population of the upper energy level and N is the ions concentration.

IV. Results and discussion

A. *Spectroscopic properties*

Starting from the absorption spectrum, the spectroscopic properties of our glass sample were determined using Judd-Ofelt theory. This required measuring the refractive index, density, and UV/Vis absorption spectrum. Results from these measurements and Judd-Ofelt analysis are presented in tables 1-4.

Table 1. Physical properties of 1 mol% Tm^{3+} -ions-doped NPZS glass

Physical properties	
Glass transition temperature, T_g (C°)	~ 250
temperature of onset crystallization, T_x (C°)	~ 400
Density, ρ (g/cm^3)	2.928
Tm^{3+} ion concentration, N (cm^{-3})	1.62×10^{20}
Reractive index, n	1.495

Fig. 1 shows the room-temperature absorption spectrum of the Tm^{3+} doped NPZS glass in the spectral range of 250-2000nm. Seven distinct absorption transitions were observed from $^3H_6$ ground state to various excited states: $^3F_4$, $^3H_5$, $^3H_4$, $^3F_3$, $^3F_2$, $^1G_4$ and $^1D_2$. These transitions occur at specific wavelengths (in nm): 1658, 1212, 792, 686, 642, 474, and 360, respectively.

The experimental oscillator strengths for all these transitions were evaluated in order to apply the Judd-Ofelt theory. Table 2 presents both the experimental and calculated oscillator strengths, along with their corresponding transitions and wavelengths. The root-mean-square (RMS) deviation between experimental and calculated values was 0,833 x 10^{-6}, indicating good agreement.

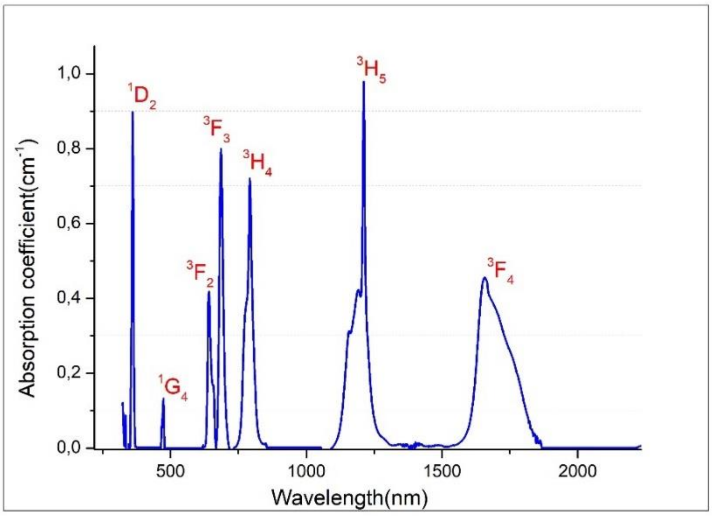

Fig. 1. Room-temperature absorption spectrum of Tm^{3+} doped NPZS glass

Table 2. Measured and calculated oscillator strength for Tm^{3+} ions in NPSZ glass

Transition: $^3H_6 \rightarrow$	λ(nm)	F_{exp}(x 10^{-6})	F_{cal}(x 10^{-6})
$^3F_4$	1658	1,447	2,023
$^3H_5$	1212	1,996	1,345
$^3H_4$	792	2,329	1,771
$^3F_3$	686	1,977	2,864
$^3F_2$	642	1,128	0,472
$^1G_4$	474	0,297	0,676
$^1D_2$	360	3,601	3,009
RMS = 0,833 x 10^{-6}			

Table 3. presents the Judd-Ofelt (JO) parameters obtained for the NPZS-glass and provides a comparison with other Tm^{3+} doped glass systems. The Judd-Ofelt parameters are significantly influenced by the host glass composition [19,23]. These parameters offer valuable information about the nature of the bond and the local environment of rare earth ions in the glass structure. Generally, $\mathbf{\Omega_2}$ is an indicator of the covalency of the rare earth-ligand bonds and the asymmetry of the local environment around the rare earth ion, and is sensitive to structural changes in the host material [24,25]. Previous studies have shown that Ω_2 increases with increasing bond valence and/or asymmetry of the rare earth ion site [26]. In contrast, Ω_4 and Ω_6 provide information about the rigidity of the host material, influenced by factors such as viscosity and dielectric constant (long-range effects). Additionally, they are affected by vibrational transitions involving the rare earth ion and its neighboring atoms [9, 19,25]. From the table 3, we can observe that NPZS-glass exhibits the lowest Ω_2 and Ω_6 values, the highest Ω_4 value, and the highest Ω_4/Ω_6 ratio compared to the other glass compositions, which suggest that the Tm^{3+} ions in the NPZS-glass have a more symmetric local environment and less covalent Tm^{3+}-ligand bonds.

Table 3. Judd-Ofelt parameters for Tm^{3+} doped NPZS glass and other glasses

Glass	Ω_2 (x10^{-20} cm^2)	Ω_4(x10^{-20} cm^2)	Ω_6(x10^{-20} cm^2)	Ω_4/Ω_6	Reference
Fluorophosphate (NPZS)	1.392	2.774	1.055	2.63	(this work)
Oxyfluoroborate (BZL)	8.37	3.20	4.34	0.73	[26]
Fluorophosphate (NPBWT)	5.28	2.32	1.16	2	[27]
Fluorophosphate (FP)	3.19	1.75	1.66	1.05	[28]
Silicate glass (TS3)	3.08	0.99	0.40	2.48	[29]

According to Jacobs-Weber theory [30], the emission intensity of rare earth ions can be assessed using the spectroscopic quality factor (Ω_4/Ω_6). This factor is crucial in predicting the performance of various laser transitions within a given host. As evident from table 3, the investigated NPZS-glass exhibits a higher spectroscopic quality factor, indication its strong potential for laser applications.

Using the Judd-Ofelt parameters, various spectroscopic parameters can be calculated, including radiative transition probabilities (A_{rad}), branching ratios (β), and radiative lifetimes (τ_{rad}). While branching ratios exhibit weak dependence on the host material, radiative transition probabilities and radiative lifetimes are more significantly influenced by the host material. Table 4. shows that NPZS glass has a longer radiative lifetime than oxyfluoroborate glass and some fluorophosphate glasses but shorter than silicate glass. The relatively long radiative lifetime could be beneficial for certain Tm^{3+} based photonic applications, particularly lasers. Typically, longer radiation lifetimes facilitate a reduction in laser oscillation thresholds, thereby improving the efficiency of laser operation [29]. However, it is important to note that laser performance can only be determined if the experimental emission cross-section and lifetime are taken into account.

Table 4. Predicted radiative transition probabilities (A_{rad}), Branching ratios (β) and radiative lifetimes (τ_{rad}) for $^3F_4 \rightarrow {}^3H_6$ transition of Tm^{3+} in NPZS-glass and other glasses

Glass	A_{rad}(s)	β(%)	τ_{rad} (ms)	Reference
Fluorophosphate (NPZS)	155.705	100	6.422	(this work)
Oxyfluoroborate	481.00	100	2.078	[26]
Fluorophosphate (NPBWT)	248.24	100	4.028	[27]
Fluorophosphate (FP)	206.50	100	4.842	[28]
Silicate glass	126.47	100	7.91	[29]

B. Laser performance parameters

The absorption and emission cross-sections are critical parameters influenced the optical gain and laser output of the active laser medium (the doped glass). Higher values of these cross-sections are desirable for efficient laser operation. The absorption cross-section $\sigma_a(\lambda)$ of Tm^{3+}ions in NPZS-glass was determined from the absorption spectrum, while the corresponding emission cross-section $\sigma_e(\lambda)$ was calculated using McCumber's theory (eq. 11). The value of $\sigma_a(\lambda)$ and $\sigma_e(\lambda)$ at $^3F_4 \rightarrow {}^3H_6$ transition of Tm^{3+} in NPZS-glass were found to be $2.81 x 10^{-21} cm^2$ and $4.22 x 10^{-21} cm^2$, respectively. Figure 2 indicates a shift of the emission cross-section towards longer wavelengths.

Using the calculated absorption and emission cross-sections, $\sigma_a(\lambda)$ and $\sigma_e(\lambda)$, the gain coefficient $\gamma(\lambda)$ was determined according to eq. (12). This calculation assumed that the active Tm^{3+} ions reside either in the ground state ($^3H_6$) or the excited state ($^3F_4$). Fig. 3 illustrates the gain coefficient for the $^3F_4 \rightarrow {}^3H_6$ transition at various population inversion ratios (p), ranging from 0 to 1 in increments of 0.2. A positive gain is achieved when the population in the upped laser level reaches about 20% indicating a relatively low laser threshold. The maximum gain coefficient is 0.68 at 1782nm. On the other hand, we observe that the gain coefficient shifts to a higher wavelength as the population inversion increases, which means that the laser wavelength changes with the increase of the pumping ratio, and this change may be a characteristic feature of the quasi-three-level laser system [31].

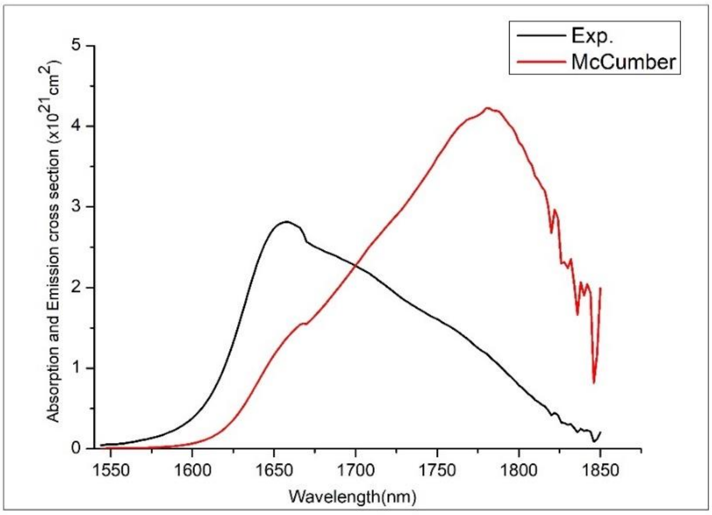

Fig. 2. Absorption and emission cross-sections of Tm^{3+} doped NPZS glass at $^3F_4 \rightarrow ^3H_6$ transition

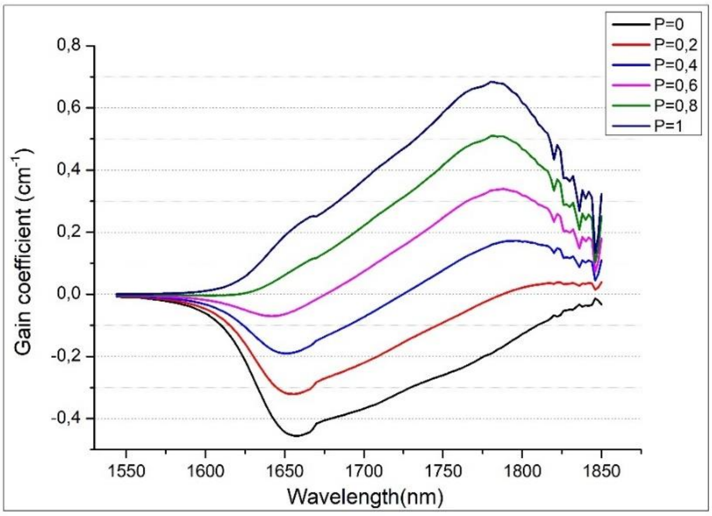

Fig. 3. Gain coefficient for the $^3F_4 \rightarrow ^3H_6$ transition of Tm^{3+} doped NPZS glass

V. Conclusion

A spectroscopic investigation on thulium doped fluorophosphate glass was conducted to evaluate its spectroscopic properties and assess its potential for laser applications. The Judd-Ofelt theory was employed to determine spectroscopic parameters, including Judd-Ofelt parameters, radiative transmission probabilities, branching ratios, lifetimes and absorption cross-section. These parameters provide crucial information about the host-dopant interaction. Analysis of results revealed that Tm^{+3} ions form relatively weak covalent bonds with nearest neighbours compared to other glass compositions. The spectroscopic quality factor (Ω_4/Ω_6) of 2.63, which expresses the emission intensity of rare earth ion, suggest strong performance potential compared to other glasses, though laser operation requires experimental verification. Using McCumber's theory, the emission cross-section was derived from absorption cross-section measurements. These data were utilized to calculate the gain coefficient for the $^3F_4 \rightarrow ^3H_6$ laser transition. The analysis demonstrated positive gain achievement at population inversion ratios exceeding 20%. Additionally, a wavelength shift in the gain coefficient was observed with increasing population inversion ratio, indicating laser wavelength dependence on pumping ratio, a characteristic potentially to quasi-three level laser system behaviour. The obtained spectroscopic properties suggest that this Tm^{+3} doped fluorophosphate glass composition presents a promising candidate for active optical applications, particularly in lasers and optical amplifier systems.

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