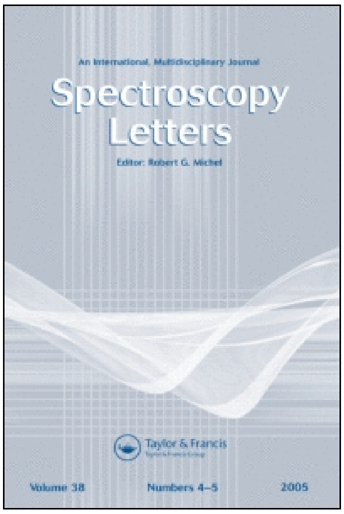

Spectroscopy Letters
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597299

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To cite this Article Reddy, B. Sudhakar , Buddhudu, S. , Rao, K. S. R. K. , Babu, P. Naresh and Annapurna, K.(2008) 'Optical Analysis of Er^{3+}:Boro-Fluoro-Phosphate Glasses', Spectroscopy Letters, 41: 8, 376 — 384
To link to this Article: DOI: 10.1080/00387010802425035
URL: http://dx.doi.org/10.1080/00387010802425035

Spectroscopy Letters, 41:376–384, 2008

ISSN: 0038-7010 print/1532-2289 online
DOI: 10.1080/00387010802425035

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Optical Analysis of Er^{3+}:Boro-Fluoro-Phosphate Glasses

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ABSTRACT This paper deals with the preparation and optical analysis of Er^{3+} (0.2 mol%) boro-fluoro-phosphate glasses in the following glass compositions:

Series A: 69.8 B_2O_3–10 P_2O_5–10($ZnO/CdO/TeO_2$)–10 AlF_3

Series B: 69.8 B_2O_3–10 P_2O_5–10($ZnO/CdO/TeO_2$)–10 LiF

Measured Vis-NIR absorption spectra of Er^{3+}:boro-fluoro-phosphate glasses have revealed *nine* absorption bands at 377 nm, 405 nm, 450 nm, 486 nm, 519 nm, 543 nm, 649 nm, 973 nm and 1529 nm, which correspond with the transitions of $^4I_{15/2} \rightarrow {}^4G_{11/2}$, ($^2G_{9/2}$,$^4H_{9/2}$), $^4F_{5/2}$, $^4F_{7/2}$, $^2H_{11/2}$, $^4S_{3/2}$, $^4F_{9/2}$, $^4I_{11/2}$, and $^4I_{13/2}$, respectively. With an excitation at $\lambda_{exci} = 375$ nm, a bright *green* emission ($^4S_{3/2} \rightarrow {}^4I_{15/2}$) at 547 nm has been observed from these erbium glasses. Judd–Ofelt characteristic intensity Ω_λ ($\lambda = 2, 4, 6$) parameters are obtained from the absorption spectra, and these results were used to compute the radiative properties of Er^{3+}:boro-fluoro-phosphate glasses. The NIR emission ($^4I_{13/2} \rightarrow {}^4I_{15/2}$) at 1547 nm from these glasses was measured with an Ar^+ laser (514.5 nm) as an excitation source.

KEYWORDS Er^{3+} glasses, optical analysis

INTRODUCTION

Glasses are attractive materials for different applications, based on their physical, electrical, and optical properties.[1–10] In recent times, there has been a great deal of work carried out on the study of luminescent properties of rare–earth doped glasses because of their potential uses in optical communications.[11–14] Among the glass hosts, silicates (SiO_2), borates (B_2O_3), and phosphates (P_2O_5) have been identified as the conventional glassy systems due to their good glass-forming ability. These glasses possess hardness, transparency, resistance toward moisture, and their refractive indices are of great importance with an extended IR transmission and good chemical durability.[15–26] Among the trivalent lanthanide ions, Er^{3+} has been an important and interesting ion, because it can yield a broad band at the third communication window.[27] We have recently reported on the physical properties and x-ray diffraction (XRD), Fourier transform infrared (FT-IR), and Differential Thermal Analysis (DTA) results of boro-fluoro-phosphate

Received 20 August 2007; accepted 26 April 2008.

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glasses.[28] We have also analyzed the spectral properties of Eu^{3+} and Tb^{3+} ions-doped boro-fluoro-phosphate glasses.[29] In the present paper, we report on the absorption and emission spectra of Er^{3+}-doped boro-fluoro-phosphate glasses by the application of the conventional Judd–Ofelt theory[30,31] to evaluate the radiative properties of the emission transitions of these glasses.

MATERIALS AND METHODS

Glasses Preparation

Two new glass series (*Series A*: B_2O_3–P_2O_5–ZnO/CdO/TeO_2–AlF_3; *Series B*: B_2O_3–P_2O_5–ZnO/CdO/TeO_2–LiF) have been prepared with the chemical compositions shown in Table 1.

The chemicals used in the current work were H_3BO_3, $(NaPO_3)_6$, Er_2O_3, ZnO, CdO, AlF_3, and LiF of reagent grade of good quality. Appropriate amounts of chemicals were weighed and mixed thoroughly in an agate mortar. Each batch, weighing about 10 g, was melted in an electric furnace at 950°C for an hour. These melts were quenched in between two brass plates to obtain 2- to 3-cm-diameter glass disks of 0.3-cm thickness each. Glasses thus obtained were annealed at 200°C for an hour to remove thermal strains, if any.

Measurements

For an Er^{3+} glass, x-ray diffraction profile was obtained on a Shimadzu (Tokyo, Japan) XD3A diffractometer with an Ni filter and CuK_α ($\lambda = 1.542$ Å) radiation with an applied voltage of 30 kV and 20 mA anode current, calibrated with Si at the rate of 2°/min. The absorption spectra of Er^{3+}:boro-fluoro-phosphate glasses were measured on a Varian-Cary-Win Spectrometer (Jasco-V570). Both the excitation and visible emission spectra of Er^{3+} glasses were measured on a SPEX Fluorolog-2 Fluorimeter with datamax software to acquire the data with a Xe arc lamp (150 W) as the excitation source.

Near Infra Red (NIR) photoluminescence spectra of the Er^{3+}:boro-fluoro-phosphate glasses were measured on a Horiba Triax-550 grating monochromator (Jobin Yvonhoriba) equipped with a liquid nitrogen (LNT)-cooled InGaAs photodetector (Electro-Optical System, Inc) in the NIR wavelength range of 1450–1700 nm that was attached to a lock-in-amplifier (SR 830 DSP; Standard Research Systems) and an Ar^+ ion laser (514.5 nm) (Lexel Model 85 Ion Laser, 5 Mw to 200 mW) as the excitation source.

TABLE 1 Chemical Compositions of Series A and Series B Classes

Glass	Chemical composition (mol%)							
	B_2O_3	P_2O_5	ZnO	CdO	TeO_2	AlF_3	LiF	Er_2O_3
Series A								
Er^{3+}:ZnO–AlF_3	69.8	10	10	—	—	10	—	0.2
Er^{3+}:CdO–AlF_3	69.8	10	—	10	—	10	—	0.2
Er^{3+}:TeO_2–AlF_3	69.8	10	—	—	10	10	—	0.2
Series B								
Er^{3+}:ZnO–LiF	69.8	10	10	—	—	—	10	0.2
Er^{3+}:CdO–LiF	69.8	10	—	10	—	—	10	0.2
Er^{3+}:TeO_2–LiF	69.8	10	—	—	10	—	10	0.2

RESULTS AND DISCUSSION

Optical Absorption Spectra

The measured XRD profile (Fig. 1) of Er^{3+}:boro-fluoro-phosphate glass has confirmed its amorphous nature. Visible-NIR absorption spectra of Er^{3+}:boro-fluoro-phosphate glasses are presented in Figs. 2a, b, with nine distinctive and sharp absorption bands at 377 nm, 405 nm, 450 nm, 486 nm, 519 nm, 543 nm, 649 nm, 973 nm, and 1529 nm, and these bands are appropriately assigned to the electronic transitions by following the literature reports.[32–34] In order to apply the Judd–Ofelt theory to these absorption spectra, well-defined absorption bands

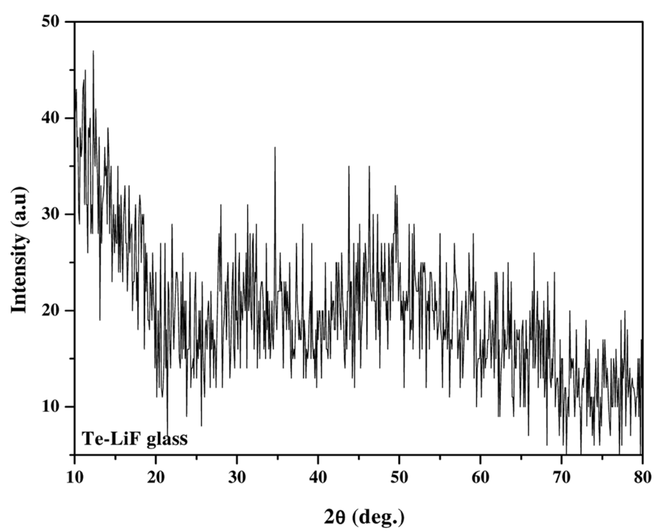

FIGURE 1 XRD profile of Er^{3+}:boro-fluoro-phosphate glass.

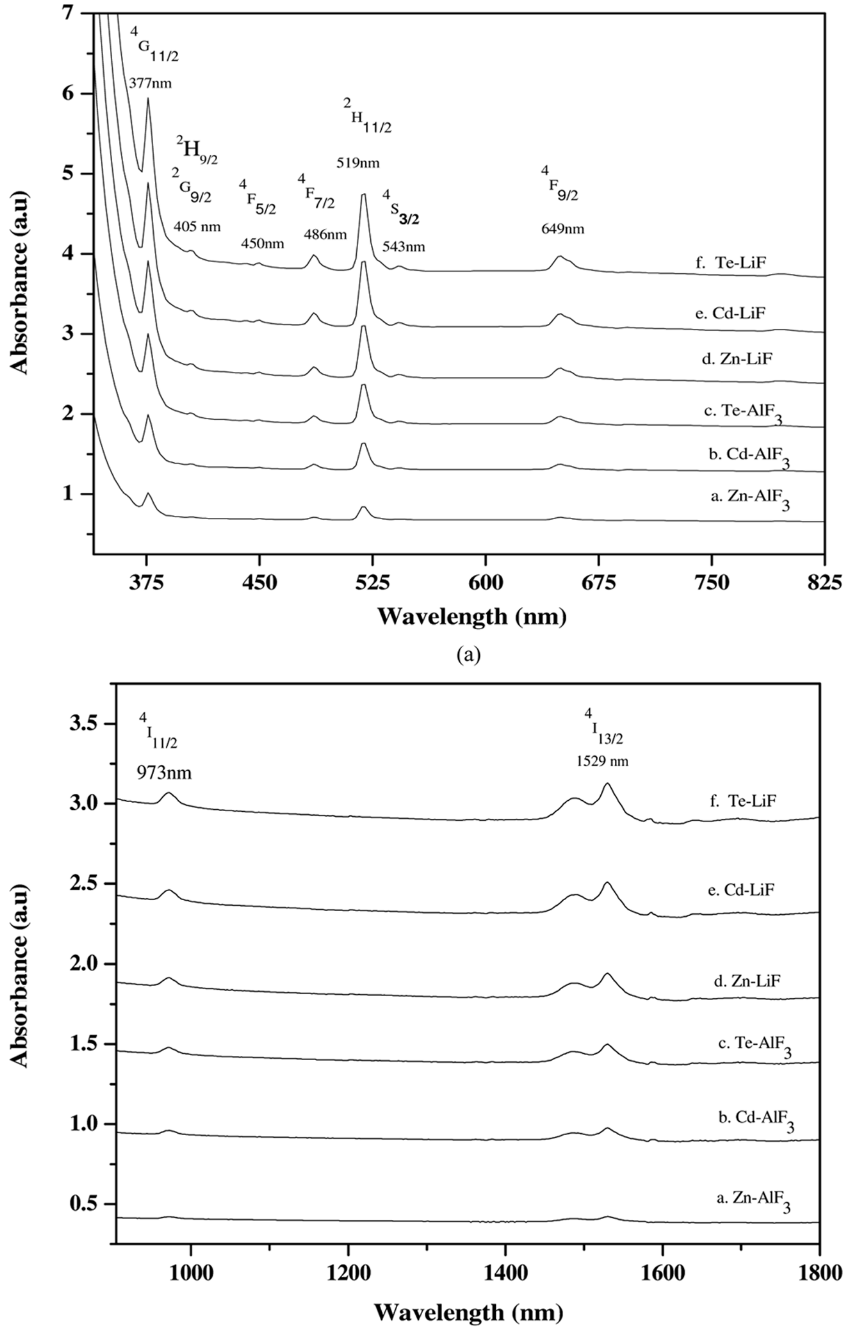

FIGURE 2 (a) Visible absorption spectra of Er^{3+}:boro-fluoro-phosphate glasses; (b) NIR absorption spectra of Er^{3+}:boro-fluoro-phosphate glasses.

such as ${}^4I_{15/2} \rightarrow {}^4G_{11/2}$, ${}^4F_{7/2}$, ${}^2H_{11/2}$, ${}^4S_{3/2}$, and ${}^4F_{9/2}$ have been considered. The Judd–Ofelt (J–O) intensity parameters[35] have been derived by performing a least squares fit analysis, and the results are presented in Table 2. The validity of the fitting has been examined by comparing the experimental and theoretical oscillator strengths of Er^{3+} absorption bands, which are also listed in the same table along with the estimated Root Mean Square (RMS) deviations. The low f_{RMS} values clearly indicate the fairness of the fitting. J–O intensity (Ω_λ) parameters have been computed for understanding the influence of glass host matrix environment on the rare-earth ions as they contain implicitly the crystal field term and radial integral wave functions. Spectral oscillator strength of ${}^4I_{15/2} \rightarrow {}^2H_{11/2}$ at 519 nm has a higher value, and this is a hypersensitive transition (HST) that follows the selection rules of $\Delta L = 2$ and

TABLE 2 Experimental and Calculated Oscillator Strengths ($f \times 10^{-6}$) of Absorption Transition. Judd–Oflet Parameters ($\Omega_\lambda \times 10^{-20}$ cm^2), and Spectroscopic Quality Factor (Ω_4/Ω_6) of Er^{3-} Ions in Boro-Fluoro-Phosphate Glasses

	Series A						Series B					
	$ZnO–AlF_3$		$CdO–AlF_3$		$TeO_2–AlF_3$		ZnO–LiF		CdO–LiF		TeO_2–LiF	
Absorption transition $^4I_{15/2} \rightarrow$	$f_{Exp} \times 10^{-6}$	$f_{Cal} \times 10^{-6}$	$f_{Exp} \times 10^{-6}$	$f_{Cal} \times 10^{-6}$	$f_{Exp} \times 10^{-6}$	$f_{Cal} \times 10^{-6}$	$f_{Exp} \times 10^{-6}$	$f_{Cal} \times 10^{-6}$	$f_{Exp} \times 10^{-6}$	$f_{Cal} \times 10^{-6}$	$f_{Exp} \times 10^{-6}$	$f_{Cal} \times 10^{-6}$
$^4I_{13/2}$	—	1.82	—	1.63	—	1.54	—	1.52	—	1.68	—	1.89
$^4I_{11/2}$	—	0.75	—	0.68	—	0.64	—	0.63	—	0.70	—	0.79
$^4I_{9/2}$	—	0.30	—	0.29	—	0.25	—	0.26	—	0.30	—	0.28
$^4F_{9/24}$	2.43	2.31	2.28	2.15	2.05	1.96	2.08	1.99	2.35	2.23	2.43	2.30
$^4S_{3/2}$	1.65	0.73	1.76	0.66	1.34	0.62	1.37	0.66	1.64	0.68	1.83	0.77
$^2H_{11/2}$	2.52	2.65	2.47	2.45	2.24	2.56	2.16	2.28	2.45	2.55	2.56	2.84
$^4F_{7/2}$	2.24	2.64	1.91	2.44	1.93	2.27	1.90	2.26	2.05	2.51	2.26	2.77
$^4F_{5/2}$	—	0.49	—	0.45	—	0.43	—	0.42	—	0.46	—	0.53
$^4F_{3/2}$	—	0.50	—	0.46	—	0.43	—	0.43	—	0.47	—	0.54
^2G, $^2H_{9/2}$	—	1.03	—	0.95	—	0.89	—	0.88	—	1.00	—	1.10
$^4G_{11/2}$	4.82	4.68	4.30	4.32	4.87	4.52	4.17	4.02	4.63	4.50	5.33	5.01
f_{RMS}	7.271×10^{-7}		8.685×10^{-7}		6.584×10^{-7}		2.758×10^{-7}		7.663×10^{-7}		8.890×10^{-7}	
J–O Parameters												
$\Omega_2 \times 10^{\times 20}$	0.957		0.865		1.075		0.822		0.922		1.187	
$\Omega_4 \times 10^{-20}$	1.325		1.257		1.128		1.168		1.356		1.240	
$\Omega_6 \times 10^{-20}$	1.780		1.630		1.573		1.526		1.719		1.962	
$\Omega_4/\Omega 6$	0.744		0.771		0.717		0.765		0.788		0.632	

TABLE 3 **Estimated Values of Spontaneous Emission Transition Probabilities (A), Radiative Rate (A_T), Radiative Lifetime (τ_R), and Branching Ratios (β%) of Emission Transitions of Er^{3+} Ions in Boro-Fluoro-Phosphate Glasses**

	Series A						Series B					
	ZnO–AlF_3		CdO–AlF_3		TeO_2–AlF_3		ZnO–LiF		CdO–LiF		TeO_2–LiF	
Emission transition	A (s^{-1})	β%	A (s^{-1})	β%	A (s^{-1})	β%	A (s^{-1})	β%	A (s^{-1})	β%	A (s^{-1})	β%
$^4I_{13/2} \rightarrow I_{15/2}$	148	100	138	100	123	100	124	100	134	100	150	100
A_T (s^{-1})	148		138		123		124		134		150	
τ_R (ms)	6.75		7.22		8.13		8.04		7.45		6.68	
$^4I_{11/2} \rightarrow {}^4I_{13/2}$	222.25	11	21	11	19	11	19	11	20	11	22	11
$^4I_{15/2}$	174.5	89	163	89	146	89	146	89	158	89	178	89
A_T (s^{-1})	196.7		184		165		165		178		201	
τ_R (ms)	5.08		5.45		6.07		6.07		5.63		4.97	
$^4F_{9/2} \rightarrow {}^4I_{9/2}$	0.8	—	0.779	—	0.8	—	0.7	—	0.7	—	0.9	—
$^4I_{11/2}$	85.01	5	29	5	71.07	5	71.3	5	76.8	5	86.8	5
$^4I_{13/2}$	68.57	4	65.26	4	56.13	4	58.4	4	64.0	4	64.1	4
$^4I_{15/2}$	1532	91	1455	91	1250	91	1303	91	1423	91	1448	91
A_T (s^{-1})	1687		1600		1378		1433		1565		1600	
τ_R (μs)	593		625		726		698		639		625	
$^4S_{3/2} \rightarrow {}^4I_{9/2}$	77.64	3	72.94	3	63.99	3	65.5	3	70.9	3	76.8	3
$^4I_{11/2}$	239.8	10	233.8	10	199.0	10	201	10	217	10	244	10
$^4I_{13/2}$	161.2	7	150.4	7	133.8	7	135	7	146	7	164	7
$^4I_{15/2}$	1939	80	1809	80	1609	80	1626	80	1751	80	1974	80
A_T (s^{-1})	2418		2256		2006		2028		2184		2459	
τ_R (μs)	414		443		499		493		458		407	
$^2H_{11/2} \rightarrow {}^4F_{9/2}$	77.5	4	72.5	4	64.5	4	65.2	4	70.4	4	77.8	4
$^4I_{9/2}$	368.9	19	344.2	19	306.9	19	309	19	333	19	376	19
$^4I_{11/2}$	320.0	16	301.8	16	275.5	16	271	16	295	16	315	16
$^4I_{13/2}$	377.5	19	359.6	19	330.9	19	323	19	355	19	359	19
$^4I_{15/2}$	822.7	42	781.8	42	670.1	42	700	42	766	42	773	42
A_T (s^{-1})	1967		1860		1648		1668		1819		1900	
τ_R (μs)	504		538		607		599		550		526	

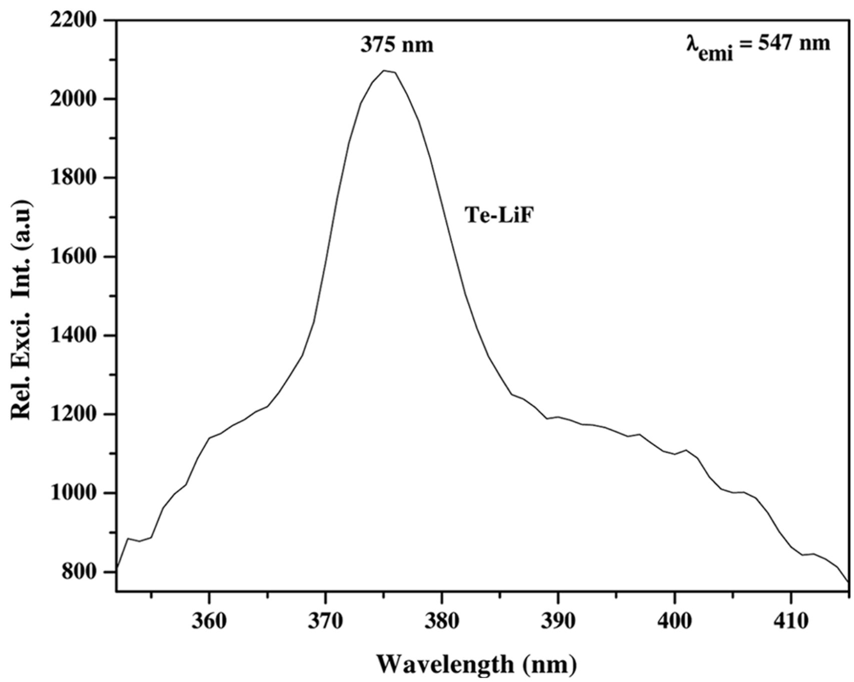

FIGURE 3 **Excitation spectrum of Er^{3+}:boro-fluoro-phosphate glass.**

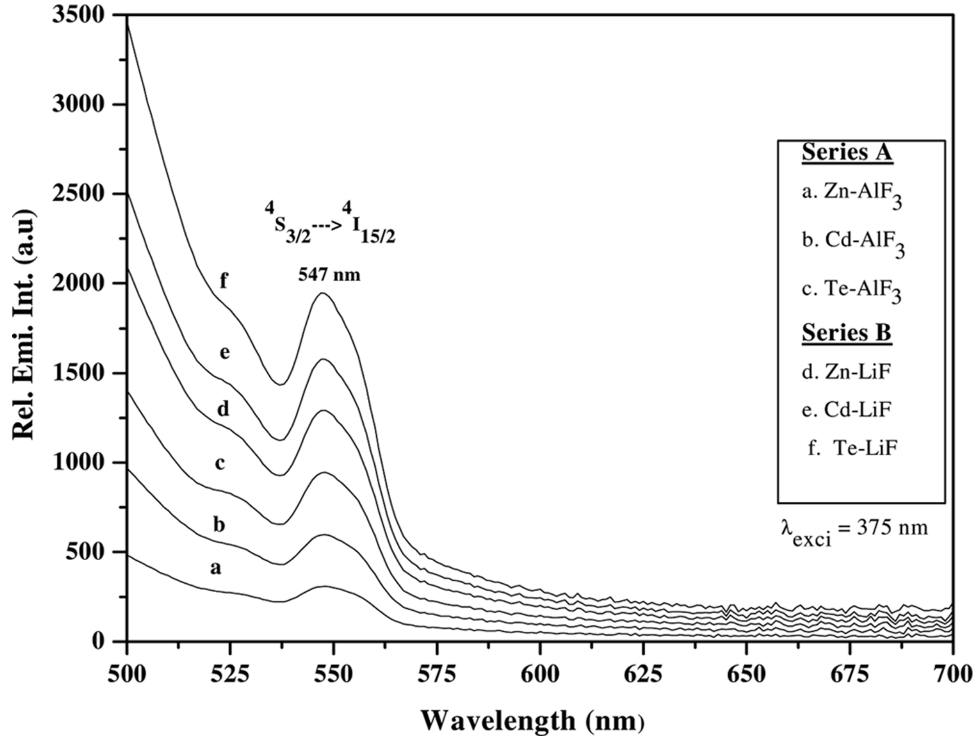

FIGURE 4 Visible emission spectra of Er^{3+}:boro-fluoro-phosphate glasses.

$\Delta J = 2$. Because it is a hypersensitive transition, it shows an intensity domination over the other transitions. For a hypersensitive transition, its intensity is more sensitive to the glass chemical composition.[36] In literature, it is considered that the Ω_2 is affected by the covalency of RE-O bond as well as the asymmetry around the rare-earth ion site, whereas Ω_4 and Ω_6 parameters are related to the covalent bonding

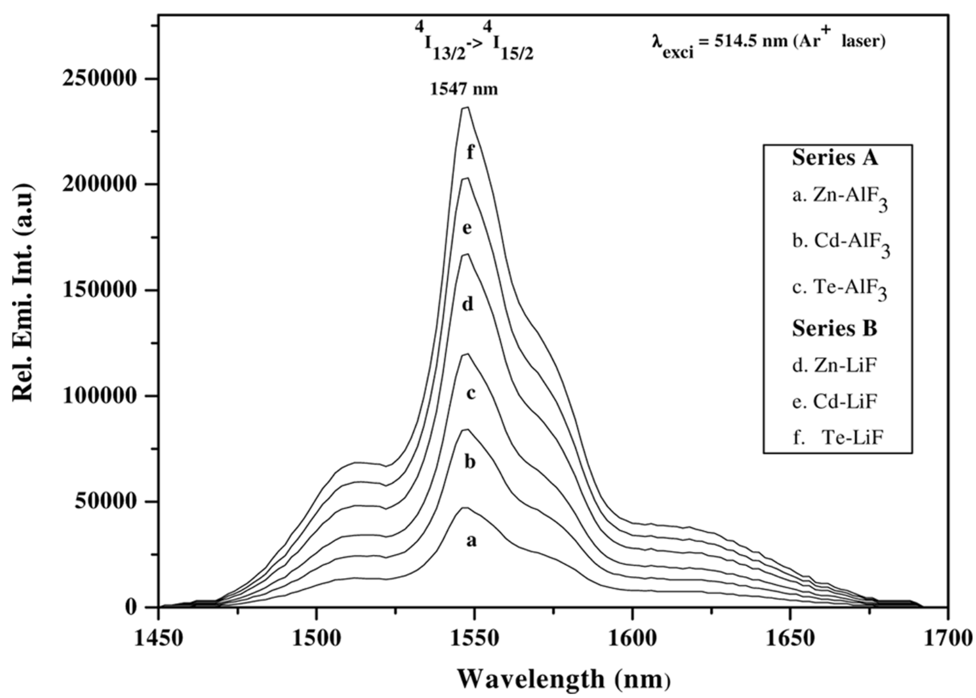

FIGURE 5 NIR emission spectra of Er^{3+}:boro-fluoro-phosphate glasses.

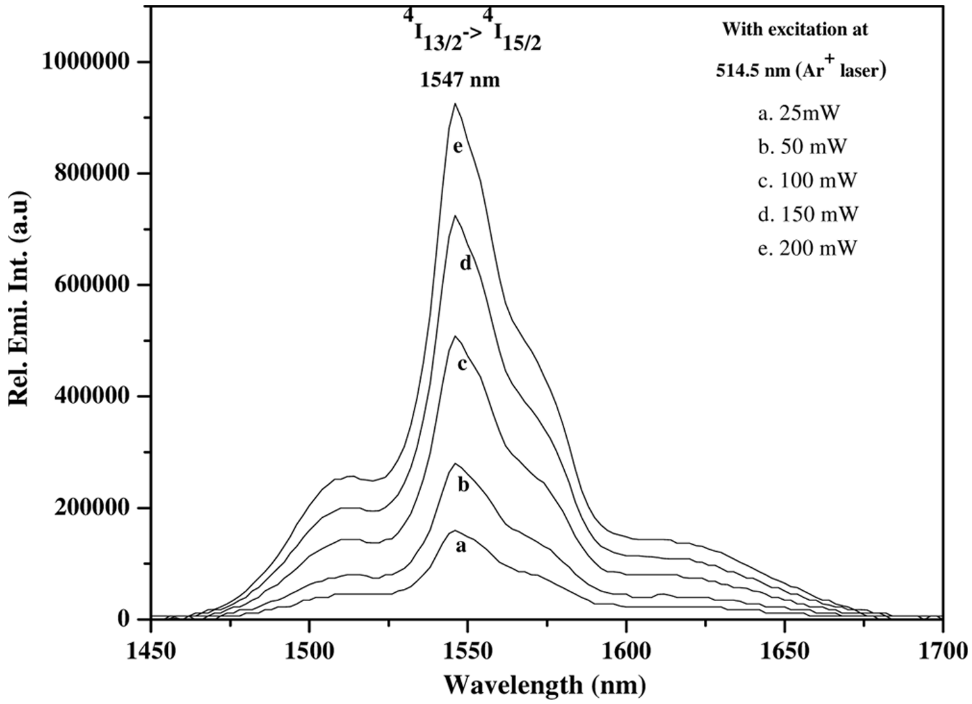

FIGURE 6 Excitation source power dependent NIR emission spectra of Er^{3+}:boro-fluoro-phosphate glasses.

only.[37–39] It is also known that Ω_2 and Ω_6 parameters could behave in a different way with regard to the covalent bonding of rare-earth ion with its ligands (O_2 or F); that is, the increasing covalency trend could also enhance the value of Ω_2 while decreasing the Ω_6 value. In all six glasses, J–O intensity parameters follow the trends of $\Omega_2 < \Omega_4 < \Omega_6$ and are found to be changing significantly with the change of the $ZnO/CdO/TeO_2$ and AlF_3/LiF respectively. The lower value of Ω_2 and higher Ω_6 could indicate a less asymmetric nature in the vicinity of Er^{3+} and with a low covalency situation from the ligands of these hosts.

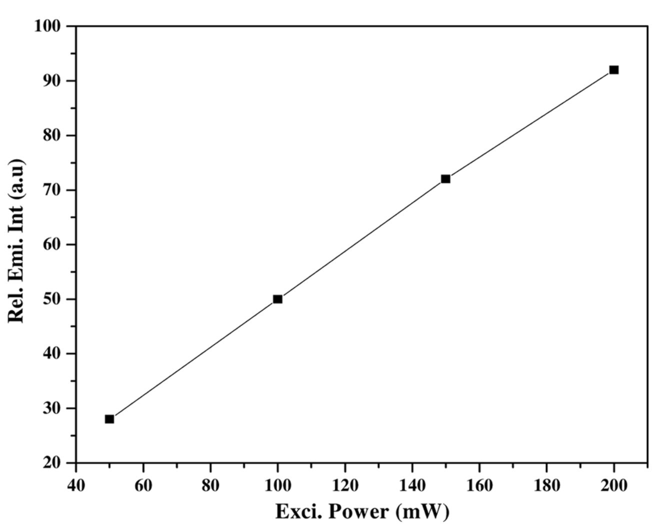

FIGURE 7 Emission (at 1547 nm) intensity linearity trend with the source power increase from Er^{3+}:boro-fluoro-phosphate glass.

Radiative Properties

By using the Judd–Ofelt intensity (Ω_λ) parameters, radiative properties of emission bands of Er^{3+}:TeO_2–LiF optical glass have been computed, and the results relating to spontaneous emission transition probability (A), radiative transition rate (ΣA_T), radiative lifetime (τ_R), and branching ratios (β%), of the emission transition $^4I_{13/2} \rightarrow {}^4I_{15/2}$ are presented in Table 3. The radiative properties of Er^{3+} glasses mainly depend on the Ω_4 and Ω_6 parameters and the ratio (Ω_4/Ω_6), which is known as a spectroscopic quality factor that describes the strength of the emission transition. For Er^{3+}: boro-fluoro-phosphate glasses, the calculated values of spectroscopic quality factor (Ω_4/Ω_6) are in the range 0.63–0.78 as presented in Table 2. Radiative properties of Er^{3+}: Boro-fluoro phosphate glasses of series A and B are presented in Tables 4 and 5 respectively. The values of transition probabilities of emission transition $^4S_{3/2} \rightarrow {}^4I_{15/2}$ are larger than the other transitions and show smaller variations in the $ZnO/CdO/TeO_2$ with AlF_3/LiF replacements in the glass compositions,

TABLE 4 **Emission Transition Peak Position (λ_P), Bandwidth ($\Delta\lambda_P$) Transition Probability (*A*), and Emission Cross Section ($\sigma_P^E \times 10^{-20}$) of Measured Emission Transition of Er^{3+} Ions in Boro-Fluro-Phosphate Glasses (Series A)**

	ZnO–AlF_3			CdO–AlF_3			TeO_2–AlF_3		
Emission transition	λ_P (nm)	$\Delta\lambda_{P(nm)}$	σ_P (cm^2)	λ_p (nm)	$\Delta\lambda_P$ (nm)	σ_P^E (cm^2)	λ_p (nm)	$\Delta\lambda_P$ (nm)	σ_P^E (cm^2)
$^4I_{13/2} \rightarrow {}^4I_{15/2}$	1547	37.6	1.15	1547	36.7	113	1547	35	1.07
$^4S_{3/2} \rightarrow {}^4I_{15/2}$	548	18.8	0.47	548	19.0	0.45	548	18.5	0.42

TABLE 5 **Emission Transition Peak Position (λ_P), Bandwidth ($\Delta\lambda_P$) Transition Probability (*A*), and Emission Cross Section ($\sigma_P^E \times 10^{-20}$) of Measured Emission Transition of Er^{3+} Ions in Boro-Fluro-Phosphate Glasses (Series B)**

	ZnO–LiF			CdO–LiF			TeO_2–LiF		
Emission transition	λ_P (nm)	$\Delta\lambda_P$ (nm)	σ_P^E (cm^2)	λ_p (nm)	$\Delta\lambda_P$ (nm)	σ_P^E (cm^2)	λ_p (nm)	$\Delta\lambda_P$ (nm)	σ_P^E (cm^2)
$^4I_{13/2} \rightarrow {}^4I_{15/2}$	1547	37.7	0.98	1547	39.4	1.04	1547	38.3	1.20
$^4S_{3/2} \rightarrow {}^4I_{15/2}$	548	20.5	0.37	548	18.1	0.46	548	19.8	0.48

but the branching ratios of the emission transitions remain the same in all six Er^{3+} glasses. The radiative lifetimes of the emission level ($^4I_{13/2} \rightarrow {}^4I_{15/2}$) of Er^{3+}:boro-fluoro-phosphate glasses are in the range 6.75–8.13 ms depending on the glass composition.

Luminescence Properties

The visible luminescence spectra of both Series A and Series B of Er^{3+}:boro-fluoro-phosphate glasses in the region 500–700 nm with an excitation at 375 nm ($^4I_{15/2} \rightarrow {}^4G_{11/2}$) are shown in Fig. 3. Because the excitation energy in this case falls in the absorption edge of the host glass itself, the excitation of erbium ion can take place partially via its ($^4I_{15/2} \rightarrow {}^4G_{11/2}$) absorption and also energy transfer from the excited state of the host. The measured visible photoluminescence spectra of Er^{3+}:boro-fluoro-phosphate glasses show strong green emission at 547 nm ($^4S_{3/2} \rightarrow {}^4I_{15/2}$). The excitation spectrum (Fig. 4) exhibits a strong excitation band at 375 nm, which indicates that the Er^{3+} ions of the glasses that are excited to $^4G_{11/2}$ level are immediately relaxed to the emitting $^4S_{3/2}$ level from where those ions could be emitting radiatively thus resulting in a green emission. Figure 5 shows an NIR emission at 1547 nm due to $^4I_{13/2} \rightarrow {}^4I_{15/2}$ transition with an Ar^+ laser (514.5 nm) excitation.[32–34] The transition $^4I_{13/2} \rightarrow {}^4I_{15/2}$ is a lasing transition with a prominent intensity with little changes on adding the ZnO/CdO/TeO_2 and AlF_3/LiF. For 0.2 mol% Er^{3+}:boro-fluoro-phosphate glasses, the excitation-dependent photoluminescence spectra are shown in Fig. 6, and it indicates that these emission peaks do not show any apparent shift with the change in excitation power. In addition, NIR emission intensity depends on excitation source power as shown in Fig. 7. The integrated PL intensity has a linear dependence on the excitation power.

CONCLUSIONS

In summary, it could be concluded that we have developed highly transparent, moisture-resistant, and brightly pink-colored Er^{3+} ion-doped boro-fluoro-phosphate glasses. By applying the Judd–Ofelt intensity parameters, radiative properties of the NIR emission ($^4I_{13/2} \rightarrow {}^4I_{15/2}$) at 1547 nm and also visible green emission ($^4S_{3/2} \rightarrow {}^4I_{15/2}$) at 547 nm have been evaluated. From both series, glasses with TeO_2 have demonstrated encouraging emission and absorption results. Thus, we could suggest 69.8 B_2O_3–10 P_2O_5–10TeO_2–10 AlF_3 from series A and 69.8 B_2O_3–10 P_2O_5–10TeO_2–10 LiF from series B with Er^{3+} ions as two novel optical glasses.

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