



Thermal stability and Judd-Ofelt analysis of optical properties of Er^{3+} -doped tellurite glasses

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Abstract: Er^{3+} -doped $\text{TeO}_2\text{--ZnO--Na}_2\text{O--B}_2\text{O}_3\text{--GeO}_2$ (TZNBG) glasses were prepared by melt-quenching method. Differential scanning calorimetry (DSC) and thermal mechanical analysis (TMA) were used to calculate thermal parameters: crystallization temperature (T_x), glass transition temperature (T_g) and thermal expansion (α). Besides, Judd-Ofelt theory is applied to analyzing absorption spectra. Intensity parameters Ω_λ ($\lambda=2, 4, 6$), transition probabilities A_{ed} , radiative lifetime τ_r , and branching ratios β of Er^{3+} transitions were obtained. Emission cross-section σ_{emis} of $^4\text{I}_{13/2} \rightarrow ^4\text{I}_{15/2}$ transition of Er^{3+} was calculated according to the theory of McCumber. All of the parameters indicate that the thermal stability and optical properties of Er^{3+} -doped TZNBG glasses are improved effectively.

Key words: tellurite glasses; thermal stability; Judd-Ofelt theory; spectroscopic properties

1 Introduction

Er^{3+} -doped tellurite glasses possess large emission cross-section, flattened broad bandwidth, excellent transmission in visible and near IR, relatively low phonon energy and a large refractive index compared with other oxide glasses [1,2]. Due to their excellent properties, the Er^{3+} -doped tellurite glasses are used as candidates for broad band amplifiers [3,4]. But, serious drawback for tellurite glasses is their relatively low thermal stability. These disadvantages result in that fibers made from these glasses are too fragile, so they do not show light guiding at all [5]. Furthermore, the tellurite glasses are hard to apply due to their drawbacks. And so far there are many studies about improving their thermal stability. Jlassi et al [6] reported that both thermal stability and quantum efficiency were improved by adding P_2O_5 to tellurite glasses. EL-MALLAWANY et al [7] made quantitative analysis of thermal properties of tellurite glass with the structure parameters like the average cross-link density, the number of bonds per unit volume, and the average stretching force constant, but the optical properties were not reported. It is necessary to possess excellent thermal stability for the tellurite glasses for further application in optical fiber amplifiers.

In this work, improving thermal stability and optical properties is the main purpose. The Er^{3+} -doped $\text{TeO}_2\text{--ZnO--Na}_2\text{O--B}_2\text{O}_3\text{--GeO}_2$ (TZNBG) glasses were elaborated by melt-quenching method. Both B_2O_3 and GeO_2 introduced into tellurite glass compositions at the same time have been rarely reported. And differential scanning calorimetry (DSC), thermal mechanical analysis (TMA), absorption and emission measurements were performed. In addition, theories of Judd-Ofelt and McCumber were applied to analyzing the optical properties.

2 Experimental

Tellurite glasses with compositions listed in Table 1 were prepared. Er_2O_3 (99.95%, mass fraction) was added into all glass samples. Na_2O and B_2O_3 were introduced in the form of Na_2CO_3 and H_3BO_3 , respectively. Batches of 15 g were prepared from commercial powders of TeO_2 (99.999%), ZnO (99.9%), Na_2CO_3 (99.9%), H_3BO_3 (99.9%) and GeO_2 (99.99%). The powders were mixed in a mortar and immersed in CCl_4 which was as a reagent of dehydration for 10 min at room temperature. The homogeneous mixture was melted in an alumina crucible at 600 °C for 0.5–1 h, then at 900 °C for 1–2 h in a furnace. When the melting was completed, the liquids

Table 1 Compositions of Er³⁺-doped tellurite glasses

Glass sample	$x(\text{TeO}_2)/\%$	$x(\text{Na}_2\text{O})/\%$	$x(\text{ZnO})/\%$	$x(\text{B}_2\text{O}_3)/\%$	$x(\text{GeO}_2)/\%$
TZN	70	10	20		
TZNBG 1	50	10	20	15	5
TZNBG 2	50	10	15	20	5
TZNBG 3	50	10	10	25	5
TZNBG 4	50	10	0	35	5

1% Er₂O₃ was added into all glass samples.

were poured into preheated massive graphite plates at 300–320 °C and annealed at this temperature for 3 h. The glasses were cooled to 100 °C after 24 h. The glass blocks prepared were cut into desired dimensions and optically polished for different measurements.

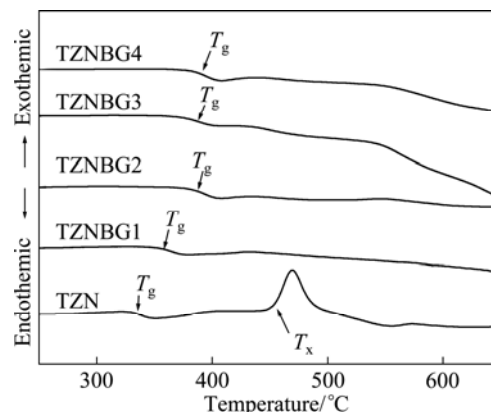
Thermal analysis was performed with a TAS100 thermal analytical instrument and Netzsch DTA 449 PC differential scanning calorimeter at a heating rate of 10 °C/min. The absorption spectra were recorded by a Perkin-Elmer Lambda-900 spectrophotometer in the wavelength range of 400–1700 nm. And emission spectra were collected by using Edinburgh Instruments Ltd FLSP 920 spectrophotometer, with 976 nm laser diode as the excitation source. Glass samples for optical and spectroscopic measurements were cut and polished in the demensions of 15 mm×15 mm×3 mm and all the optical measurements were carried out at room temperature.

3 Results and discussion

3.1 Thermal stability

In order to evaluate the thermal stability of glass samples, measurements of DSC and TMA were performed. Figure 1 presents the DSC curve of TZNBG glasses. The DSC curve shows glass transition temperature (T_g) and crystallization temperature (T_x) in glass sample. And thermal expansion coefficient α was obtained by TMA. Results of thermal parameters are listed in Table 2. ΔT is identified as: $\Delta T = T_x - T_g$. And the ΔT has been frequently used as a rough measure of the glass thermal stability [8]. Since fiber fabricating is a reheating process, any crystallization during the process will lead to more scattering loss of the fiber and then damage the optical properties. To achieve a large range of working temperature during the fiber fabricating and to obtain glass fiber with superior optical properties, it is desired that ΔT of glass as large as possible [9]. The T_g and glass softening temperature (T_f) increase for the B₂O₃ and GeO₂ introduced into glass compositions. And the crystallization peaks were extremely weak in samples of TZNBG glass. Furthermore, the value of ΔT is larger than 134 °C. In addition, α of TZNBG glass samples is in

the range of $(10.938\text{--}12.279) \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$. It is much lower than $15.466 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ of TZN glass sample. So, it suggests that introducing B₂O₃ and GeO₂ into tellurite glasses can improve their thermal stability. The reason for the performance change is structure of glass changed after introducing B₂O₃ and GeO₂ into the tellurite glasses [4].

**Fig. 1** DSC curves of glass sample**Table 2** Thermal parameters of different glass samples

Glass sample	$T_g/^\circ\text{C}$	$T_x/^\circ\text{C}$	$T_f/^\circ\text{C}$	$\alpha/(10^{-6} \text{ } ^\circ\text{C}^{-1})$	$\Delta T/^\circ\text{C}$
TZN (Present work)	335	469	353	15.466	134
TZNBG 1 (Present work)	363	*	388	12.279	—
TZNBG 2 (Present work)	388	*	423	10.938	—
TZNBG 3 (Present work)	380	*	416	11.479	—
TZNBG 4 (Present work)	392	*	421	11.286	—
TZNE 1 [6]	316	456			140
BT-70 [10]	623	715			92
BT-60 [10]	625	700			75
BT-50 [10]	592	670			78

“*” indicates that DSC curve of glass sample has no obvious crystallization peak.

3.2 Absorption spectra and Judd-Ofelt analysis

The TZNBG 2 glass sample was selected for further optical studies due to its relatively good thermal stability, and the TZN sample was as comparison.

Figure 2 illustrates the absorption spectra of Er³⁺-doped TZNBG 2 and TZN glass samples. The absorption spectra consist of eight absorption bands at 1531, 978, 796, 652, 544, 521, 488 and 451 nm, corresponding to the absorption from the ground state ⁴I_{5/2} to the excited states of ⁴I_{13/2}, ⁴I_{11/2}, ⁴I_{9/2}, ⁴F_{9/2}, ⁴S_{3/2}, ²H_{11/2}, ⁴F_{7/2} and ⁴F_{5/2} of Er³⁺, respectively. As we can see the peak position of each transition remains unchanged with adding the B₂O₃ and GeO₂.

The theory of Judd-Ofelt [11,12] is often used to calculate the spectroscopic parameters such as oscillator coefficient f , intensity parameters Ω_λ ($\lambda=2, 4, 6$),

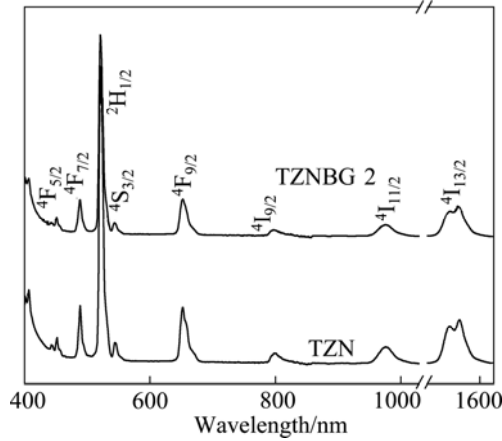


Fig. 2 Absorption spectra of Er³⁺-doped tellurite glasses

transition probability A_{ed} , branching ratio β , radiative lifetime τ_i . The oscillator coefficient for each band is computed by the following expression:

$$f_{exp} = \frac{2303mc^2}{N\pi c^2} \int \varepsilon(\sigma) d\sigma = 4.318 \times 10^{-9} \int \varepsilon(\sigma) d\sigma \quad (1)$$

where N is the number of rare earth ions per unit volume; $\varepsilon(\sigma)$ is the molar absorptivity in L/(mol·cm) of band at a mean energy σ in cm⁻¹, which is computed from the measured absorbance for known concentrations N_0 of the Er in the glass [13].

According to the Judd-Ofelt theory the oscillator strength of an electric dipole transition ($S, L, J \rightarrow S', L', J'$) is determined from formula (2):

$$f_{cal} = \frac{8\pi^2 mc}{3h(2J+1)} \frac{(n^2+2)^2}{9n} \sigma \cdot \sum_{\lambda=2,4,6} \Omega_{\lambda} \left| \left\langle (SLJ) \parallel U^{(\lambda)} \parallel (S'L'J') \right\rangle \right|^2 \quad (2)$$

where h is the Planck's constant; c is the speed of light; m is the mass of electron; n is the refractive index; $U(\lambda) = \left\langle (SLJ) \parallel U^{(\lambda)} \parallel (S'L'J') \right\rangle$ is the doubly reduced unit tensor operator that is taken from Ref. [13]. The Judd-Ofelt parameters Ω_{λ} ($\lambda=2, 4, 6$) are obtained by a least-square method and the oscillator strength f for any transition is evaluated from formula (2). The quality of the fitting of the theoretical oscillator strength values to the measured ones can be expressed by the root-mean-square δ_{rms} , which is calculated by:

$$\delta_{rms} = \left[\frac{\sum (f_{cal} - f_{exp})^2}{N_{bands} - 3} \right]^{1/2} \quad (3)$$

where N_{bands} regards the number of transition bands analyzed.

The values of Ω_{λ} ($\lambda=2, 4, 6$) can be applied to calculating the radiative transition probabilities A_{ed} ($J' \rightarrow J''$), for excited levels of rare earth ions from an

initial state J' to a final ground state J'' , is given by the following formula:

$$A_{ed}(J' \rightarrow J'') = \frac{64\pi^4 e^2 \sigma^3}{3h(2J'+1)} \frac{n(n^2+2)^2}{9} \cdot \sum_{\lambda=2,4,6} \Omega_{\lambda} \left| \left\langle (S'L'J') \parallel U^{(\lambda)} \parallel (S''L''J'') \right\rangle \right|^2 \quad (4)$$

The total transition probability (A_T) has been evaluated from $A_T = \sum A_{ed}$. The branching ratio can be identified as $\beta = A_{ed}/A_T$. The radiative lifetime τ_i was calculated from $\tau_i = 1/A_T$. The oscillator strength f and the intensity parameter (Ω_{λ}) are set out in Table 3, and radiative transition probability (A_{ed}), branching ratio (β) and the radiative lifetime (τ_i) are listed in Table 4.

Table 3 Measured (f_{mea}) and calculated (f_{cal}) oscillator coefficient and intensity parameter (Ω_{λ}) for Er³⁺-doped tellurite glasses by Judd-Ofelt theory

Transition	λ/nm	TZN		TZNBG 2	
		$f_{mea}/10^{-6}$	$f_{cal}/10^{-6}$	$f_{mea}/10^{-6}$	$f_{cal}/10^{-6}$
$^4I_{15/2} \rightarrow ^4I_{13/2}$	1531	1.332	1.215	0.961	0.879
$^4I_{15/2} \rightarrow ^4I_{11/2}$	978	0.450	0.611	0.333	0.440
$^4I_{15/2} \rightarrow ^4I_{9/2}$	796	0.239	0.225	0.220	0.154
$^4I_{15/2} \rightarrow ^4F_{9/2}$	652	1.656	1.611	1.140	1.134
$^4I_{15/2} \rightarrow ^4S_{3/2}$	544	0.348	0.465	0.221	0.341
$^4I_{15/2} \rightarrow ^2H_{11/2}$	521	7.929	7.932	5.432	5.426
$^4I_{15/2} \rightarrow ^4F_{7/2}$	488	1.258	1.764	0.921	1.272
$^4I_{15/2} \rightarrow ^4F_{5/2}$	451	0.255	0.566	0.205	0.415
$\delta_{rms}/10^{-7}$		2.855		2.023	
$\Omega_2/10^{-20}cm^2$		5.757		5.018	
$\Omega_4/10^{-20}cm^2$		1.106		0.949	
$\Omega_6/10^{-20}cm^2$		1.299		1.212	
Trend		$\Omega_2 > \Omega_6 > \Omega_4$		$\Omega_2 > \Omega_6 > \Omega_4$	
$N_0/10^{22}cm^{-3}$		2.352		1.404	

According to the theory of JACOBS and WEBER [14], erbium emission intensity can be characterized by Ω_4 and Ω_6 parameters. The smaller value of Ω_4/Ω_6 corresponds to higher intensity of laser transition $^4I_{13/2} \rightarrow ^4I_{15/2}$ of Er³⁺ [15]. In the present work, the value of Ω_4/Ω_6 is estimated to be 0.783 for Er³⁺ in TZNBG 2 glass sample, which indicates that the transition $^4I_{13/2} \rightarrow ^4I_{15/2}$ is more efficient than that in other glass samples [6,10,10]. The values of Ω_4/Ω_6 of different glass samples are included in Table 5.

3.3 Emission spectra and emission cross-section

Figure 3 exhibits the emission spectra for the $^4I_{13/2} \rightarrow ^4I_{15/2}$ transition of Er³⁺-doped TZN and TZNBG 2 glass samples. According to the McCumber theory [17], the emission cross-section σ_{emis} can be determined by

Table 4 Radiative transition probability (A_{ed}), branching ratio (β) and radiative lifetime (τ_i) of energy levels of Er^{3+} -doped in tellurite glasses

Transition	TZN				TZNBG 2			
	Average frequency/ cm^{-1}	$A_{\text{ed}}/\text{ns}^{-1}$	τ_i/ms	$\beta/\%$	Average frequency/ cm^{-1}	$A_{\text{ed}}/\text{ns}^{-1}$	τ_i/ms	$\beta/\%$
$^4\text{I}_{13/2} \rightarrow ^4\text{I}_{15/2}$	6545	129.903	7.698	1.000	6552	84.709	11.805	1.000
$^4\text{I}_{11/2} \rightarrow ^4\text{I}_{15/2}$	10232	159.641	5.544	0.885	10225	102.026	8.681	0.886
$\rightarrow ^4\text{I}_{13/2}$	3686	20.721		0.115	3673	13.165		0.114
$^4\text{I}_{9/2} \rightarrow ^4\text{I}_{15/2}$	12471	87.414	9.094	0.795	12532	52.823	15.005	0.793
$\rightarrow ^4\text{I}_{13/2}$	5925	19.734		0.179	5980	12.029		0.181
$\rightarrow ^4\text{I}_{11/2}$	2239	2.818		0.026	2308	1.791		0.027
$^4\text{F}_{9/2} \rightarrow ^4\text{I}_{15/2}$	15303	941.410	0.722	0.680	15322	590.610	1.151	0.680
$\rightarrow ^4\text{I}_{13/2}$	8757	308.299		0.223	8770	193.507		0.223
$\rightarrow ^4\text{I}_{11/2}$	5071	103.380		0.075	5098	65.369		0.075
$\rightarrow ^4\text{I}_{9/2}$	2832	32.238		0.023	2790	19.581		0.023
$^4\text{S}_{3/2} \rightarrow ^4\text{I}_{15/2}$	18320	389.280	1.479	0.576	18372	258.421	2.225	0.575
$\rightarrow ^4\text{I}_{13/2}$	11775	160.809		0.238	11820	106.969		0.238
$\rightarrow ^4\text{I}_{11/2}$	8089	75.886		0.112	8147	50.820		0.113
$\rightarrow ^4\text{I}_{9/2}$	5850	39.686		0.059	5840	26.108		0.058
$\rightarrow ^4\text{F}_{9/2}$	3018	10.563		0.016	3050	7.121		0.016
$^2\text{H}_{11/2} \rightarrow ^4\text{I}_{15/2}$	19195	7293.147	0.075	0.550	19203	4282.595	0.129	0.551
$\rightarrow ^4\text{I}_{13/2}$	12650	3167.365		0.239	12651	1858.777		0.239
$\rightarrow ^4\text{I}_{11/2}$	8964	1590.403		0.120	8978	936.158		0.120
$\rightarrow ^4\text{I}_{9/2}$	6725	895.060		0.067	6671	516.771		0.066
$\rightarrow ^4\text{F}_{9/2}$	3893	299.951		0.023	3881	174.897		0.022
$\rightarrow ^4\text{S}_{3/2}$	875	15.155		0.001	831	8.021		0.001
$^4\text{F}_{7/2} \rightarrow ^4\text{I}_{15/2}$	20449	1840.397	0.280	0.515	20501	1200.092	0.429	0.514
$\rightarrow ^4\text{I}_{13/2}$	13904	850.797		0.238	13950	555.608		0.238
$\rightarrow ^4\text{I}_{11/2}$	10218	459.479		0.129	10277	301.545		0.129
$\rightarrow ^4\text{I}_{9/2}$	7979	280.157		0.078	7969	181.327		0.078
$\rightarrow ^4\text{F}_{9/2}$	5147	116.580		0.033	5179	76.589		0.033
$\rightarrow ^4\text{S}_{3/2}$	2129	19.948		0.006	2130	12.948		0.033
$\rightarrow ^2\text{H}_{11/2}$	1254	6.920		0.002	1298	4.814		0.002
$^4\text{F}_{5/2} \rightarrow ^4\text{I}_{15/2}$	22110	690.752	0.682	0.471	22163	458.027	1.028	0.471
$\rightarrow ^4\text{I}_{13/2}$	15564	342.306		0.234	15612	227.254		0.234
$\rightarrow ^4\text{I}_{11/2}$	11878	199.369		0.136	11939	132.902		0.137
$\rightarrow ^4\text{I}_{9/2}$	9639	131.286		0.090	9631	86.491		0.089
$\rightarrow ^4\text{F}_{9/2}$	6807	65.478		0.045	6841	43.640		0.045
$\rightarrow ^4\text{S}_{3/2}$	3789	20.291		0.014	3792	13.405		0.014
$\rightarrow ^2\text{H}_{11/2}$	2914	12.002		0.008	2961	8.173		0.008
$\rightarrow ^4\text{F}_{7/2}$	1660	3.896		0.003	1662	2.576		0.003

Table 5 Value of Ω_4/Ω_6 of different glass samples

Glass sample	Ω_4/Ω_6
TZN (Present work)	0.851
TZNBG 2 (Present work)	0.783
TZNE 1 [6]	0.99
BT-70 [10]	2.09
BT-60 [10]	2.27
TW 1 [16]	2.03
TW 3 [16]	1.87
TCW 1 [16]	2.08
TCW 3 [16]	2.11

$$\sigma_{\text{emis}} = \frac{\lambda_p^4}{8\pi c n^2 \Delta\lambda_{\text{eff}}} A_{\text{ed}}(J' \rightarrow J'') \quad (5)$$

where λ_p is the peak fluorescence wavelength, and $\Delta\lambda_{\text{eff}}$ is the effective line-width of emission band, which can be calculated using the following equation:

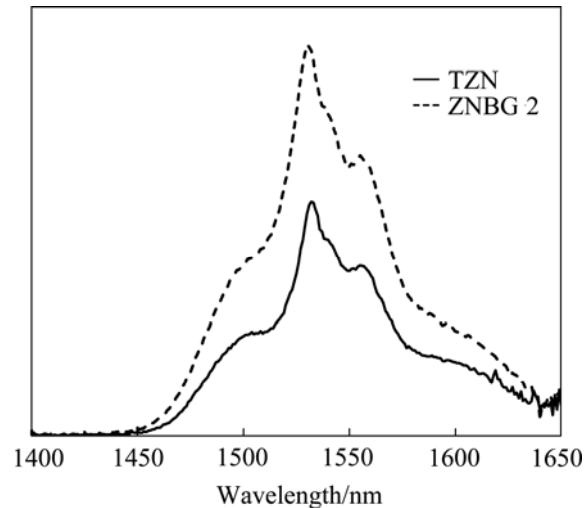


Fig. 3 Emission spectra of $^4\text{I}_{13/2} \rightarrow ^4\text{I}_{15/2}$ transition of Er^{3+} in all glasses under 976 nm excitation

$$\Delta\lambda_{\text{eff}} = \frac{\int I(\lambda)d\lambda}{I_{\text{max}}} \quad (6)$$

where I_{max} is the maximum intensity at the fluorescence emission peak.

Table 6 gives the emission cross-section σ_{emis} , and the full width at half maxima (FWHM) of the emission peak of ${}^4\text{I}_{13/2} \rightarrow {}^4\text{I}_{15/2}$ transition of Er^{3+} in different glass matrices. Bandwidth properties of the optical amplifier can be evaluated from the product of σ_{emis} and FWHM, and the larger the better. The value of $\sigma_{\text{emis}}\tau_i$ can be applied to evaluating the gain of bandwidth [16]. As the results present in Table 6, these parameters of TZNBG 2 glass sample are more excellent than those of other glass sample. Further more, the value of $\sigma_{\text{emis}} \times \text{FWHM}$ of tellurite glass samples that introduced B_2O_3 and GeO_2 is larger than TZN glass samples too [7,17]. On the other hand, the product of σ_{emis} and τ_i is the largest among these glass samples. So, the tellurite glass samples contain B_2O_3 and GeO_2 are more suitable to be used as candidate for broad band optical amplifiers.

Table 6 Emission cross-section σ_{emis} , FWHM and radiative lifetime τ_i of ${}^4\text{I}_{13/2} \rightarrow {}^4\text{I}_{15/2}$ transition of Er^{3+} in different glass samples

Glass sample	FWHM/ nm	$\sigma_{\text{emis}}/(10^{-21}\text{cm}^2)$	$\sigma_{\text{emis}} \times \text{FWHM}/(10^{-21}\text{cm}^2 \cdot \text{nm})$	$\sigma_{\text{emis}}\tau_i/(10^{-21}\text{cm}^2 \cdot \text{ms})$
TZN (Present work)	52	3.984	207.168	30.669
TZNBG 2 (Present work)	58	8.151	472.758	108.474
TZNE 1[6]	58	2.404	139.43	9.469
70TeO ₂ - 15ZnO- 15Na ₂ O- 0.1Er ₂ O ₃ [15]			332.7	0.0037
5Na ₂ O- 20Sb ₂ O ₃ - 35B ₂ O ₃ - 39SiO ₂ - 1Er ₂ O ₃ [18]	88	6.8	598.4	25.976

4 Conclusions

1) The results of DSC and thermal mechanical analysis reveal that Er^{3+} -doped TZNBG glasses possess high thermal stability, the ΔT is higher than 134 °C and the α is in the range of $(10.938\text{--}12.279) \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$.

2) The value of Ω_4/Ω_6 is estimated to be 0.783 for Er^{3+} in TZNBG glass sample, which indicates that the

transition ${}^4\text{I}_{13/2} \rightarrow {}^4\text{I}_{15/2}$ is more efficient than that in other glass samples. The emission cross-section σ_{emis} , and FWHM of 1530 nm emission peak of ${}^4\text{I}_{13/2} \rightarrow {}^4\text{I}_{15/2}$ transition of the Er^{3+} suggest that the TZNBG glass samples have good bandwidth properties and high gain of bandwidth.

3) On the basis of all the data obtained, the thermal stability and optical properties of Er^{3+} -doped TZNBG glasses are improved.

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掺铒碲酸盐的热稳定性和 Judd-Ofelt 理论分析

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摘 要：利用熔融法制备掺铒 $\text{TeO}_2\text{-ZnO-Na}_2\text{O-B}_2\text{O}_3\text{-GeO}_2$ 碲酸盐玻璃。采用差热扫描分析法(DSC)和热分析(TMA)得到玻璃的玻璃转化温度(T_g)、玻璃析晶温度(T_x)、玻璃软化温度(T_f)和热膨胀系数(α)，应用 Judd-Ofelt 理论计算玻璃中 Er^{3+} 的振子强度 Ω_λ ($\lambda=2, 4, 6$)，跃迁几率 A_{ed} ，荧光分支比 β ，辐射寿命 τ_i 。根据 McCumber 理论计算 Er^{3+} 离子 $^4\text{I}_{13/2}\rightarrow^4\text{I}_{15/2}$ 的受激发射截面 σ_{emis} 和荧光半高宽 FWHM。得出此体系的玻璃具有高热稳定性和低热膨胀性，具有较高的 Er^{3+} 离子 $^4\text{I}_{13/2}\rightarrow^4\text{I}_{15/2}$ 能级跃迁效率和较好的增益带宽性能。

关键词：碲酸盐玻璃；热稳定性；Judd-Ofelt 理论；光谱性能

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