Efficient emission of 2.7 μm from Diode-pumped Er^{3+}/Nd^{3+} Co-doped Bismuth Germanate Glass

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Abstract Mid-infrared emission spectroscopic property of Er^{3+}/Nd^{3+} co-doped bismuth germanate glasses pumped by 808 nm excitation is discussed. The absorption transition parameters of Er^{3+} are estimated by Judd-Ofelt theory. For the luminescence of 2.7 μm, the as-made glasses is qualified with higher spontaneous transition probability (58.46 s^{-1}) and larger calculated emission cross section (8.34×10^{-21} cm^2). The large energy transfer probability rate between Er^{3+} and Nd^{3+} indicate that the co-doping Nd^{3+} greatly enhances the 2.7 μm emission of Er^{3+}, and this might indicate that this glass is potentially applicable materials for mid-infrared laser devices.

Key words materials; glass; spectroscopic properties; 2.7 μm emission; bismuth germanate glasses; Er^{3+}/Nd^{3+} co-doped

OCIS codes 160.2750; 160.4760; 160.5690

Er^{3+}/Nd^{3+}共掺铋锗酸盐玻璃2.7 μm光谱性质研究

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摘要 用高温熔融法制备了Er^{3+}/Nd^{3+}共掺铋锗酸盐玻璃,研究了玻璃的吸收光谱和在808 nm抽运下的荧光光谱。研究表明:对应于2.7 μm发光,Er^{3+}/Nd^{3+}共掺铋锗酸盐玻璃具有大的自发辐射几率(58.46 s^{-1})和受激发射截面(8.34×10^{-21} cm^2)。通过对铒钕离子之间能量传递微观系数和效率的计算得知,钕离子不但可以增加铒离子2.7 μm发光上能级的布局数,而且可以减小其下能级的布局数,良好的2.7 μm发光性质表明Er^{3+}/Nd^{3+}共掺铋锗酸盐玻璃是一种潜在的中红外发光激活介质。

关键词 材料; 玻璃; 光谱性质; 2.7 μm发光; 铋锗酸盐玻璃; Er^{3+}/Nd^{3+}共掺

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1 Introduction

Light sources with 3 μm emission rate are progressing rapidly due to their wide range applications, including remote sense, environmental pollution monitoring, laser radar, medical diagnostics, etc[1-2]. Recently, laser glasses doped with Er^{3+} has been intensely studied because of its easy fabrication process and reshaping operation in mid-IR region[3-4]. Many researchers have devoted their efforts to exploit fluoride glasses with low phonon energy[5]. Compared with fluoride glasses, oxide glasses are easily prepared, with good thermal stability and chemical durability, as well as good physical and mechanical performance, though its phonon energy is large[2,6]. Usually, 2.7 μm emissions can't be efficiently obtained in Er^{3+}-doped glasses since the lifetime of the emitting level Er^{3+}:$^4I_{11/2}$ is considerably shorter than that of the terminal

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level[3]. To overcome this problem, co-doping of Nd^{3+} with Er^{3+} is used as a feasible alternative to depopulate the lower level[4].

To our knowledge, Er^{3+}/Nd^{3+} co-doped method has been adopted in chalcohalide[7], fluorophosphates (FP)[8-9], tellurite glasses[4] and fluorotellurite glasses[10]. Bismuth germanate glasses is proved to be an excellent material due to its easier preparation process, good physical stability and chemical durability than chalcohalide[4,11]. Compared with tellurite and FP glasses, bismuth germanate glasses is characterized with significantly low phonon energy, high refractive index and mid-IR optical transparency[11]. The high refractive index and mid-IR optical transparency will benefit the high stimulated emission cross section and good mid-IR emission properties, respectively. The low phonon energy can reduce the multi-phonon relaxation rate of the excited state. However, reports concerning the 2.7 μm emission in Er^{3+}/Nd^{3+} co-doped bismuth glasses are not so common. In this paper, the spectroscopic properties and energy transfer mechanism in Er^{3+}/Nd^{3+} co-doped bismuth germanate glasses are presented. The experimental results demonstrate that this glass system has many advantages, such as 2.7 μm radiative characteristics over other Er^{3+}/Nd^{3+} co-doped glasses.

2 Experimental

The two samples with compositions (cationic fraction, %): $55BiO_{1.5}$-$30GeO_2$-$15NaO_{0.5}$-$xErO_{1.5}$-$yNdO_{1.5}$ (x=0, y=2; x=2, y=0; x=2, y=2) are prepared using highly-pure Bi_2O_3, GeO_2, Na_2CO_3, Er_2O_3 and Nd_2O_3 powder as starting materials. The well-mixed raw materials for 30 g batch were placed in an alumina crucible. The samples were melted at 1050 ℃ for 30 min under oxygen atmosphere. Dry oxygen gas was introduced in order to remove hydroxyl groups. The melts were then collected on preheated stainless-steel mold and annealed for several hours near the T_g (glass transition temperature). The annealed sample was cut and polished to the size of 20 mm×10 mm×1 mm for optical measurements.

The refractive index was measured using a Metricon Model 2010/M Prism Coupler. The mid-infrared transmission spectrum ranging from 2.5 to 7.0 μm was measured by a Thermo Nicolet (Nexus FT-IR Spectrometer). Room temperature absorption spectra were obtained with a Perkin-Elmer Lambda 900UV/VIS/NIR spectrophotometer with 1 nm steps. Fluorescence spectra were tested by TRIAX550 spectrophotometer under the excitation of 808 nm laser diode source. The fluorescence decay curves were measured pumped by an 808 nm laser diode using FLSP 920 (Edinburgh instruments Ltd., UK) instrument. Spectra are recorded at room temperature.

3 Results and discussions

3.1 Mid-IR transmission and UV-Vis-NIR absorption spectra

Fig.1 (a) shows the infrared transmittance spectra of Er^{3+}-, Nd^{3+}- and Er^{3+}/Nd^{3+} doped samples ranging from 2.5 to 7 μm . The IR absorption cut-off edge of these glasses is 6.5 μm, which is larger than that of FP glasses (4.2 μm)[12]. It is noticed that the maximum transmittance at 2.7 μm band reached 84% in codoped glass. The small absorption band at 3 μm is typically for H_2O absorption. Hydroxyl group absorption is catastrophic for some infrared applications and quantum efficiency[3], so it would be better that the applied glass in the mid-IR region have lower hydroxyl group content. Hydroxyl group concentration can be estimated from the absorption coefficient (α_{OH}) at 3 μm

$$\alpha_{OH} = \frac{\ln(T_0/T)}{l}, \tag{1}$$

where T_0 is the transmittance at the baseline, T is the transmittance around 3 μm region and l is the sample thickness. As shown in Fig.1, the prepared three samples exhibit the low concentration of the hydroxyl group even though these values are different from each other. The calculated absorption coefficient of codoped sample is 0.43 cm^{-1}, which is smaller than that of tellurite glasses (2.1 cm^{-1})[13]. The good infrared transmission characteristics of this glass are favorable because of its spectroscopic properties in the mid-infrared region.

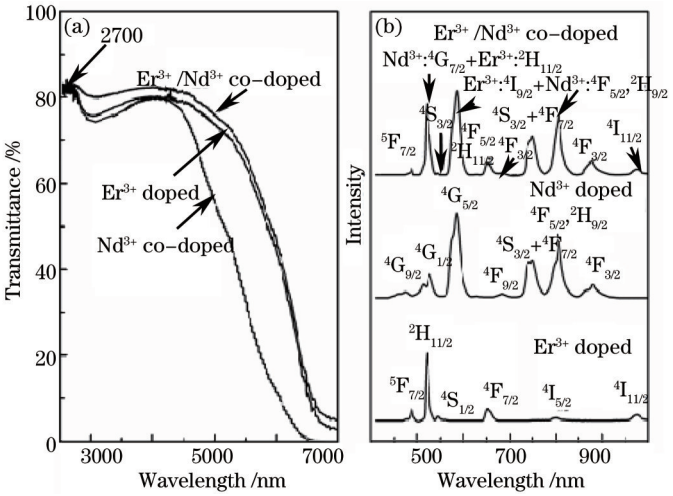

Fig.1 (a) FTIR transmittance spectra of Er^{3+}-、Nd^{3+}- and Er^{3+}/Nd^{3+}-doped bismuth germanate glass (1mm thickness); (b) absorption spectra of Er^{3+}-、Nd^{3+}- and Er^{3+}/Nd^{3+}-doped bismuth germanate glasses

The room temperature absorption spectra of the three samples in 400~1000 nm region are shown in Fig.1 (b) and the absorption band attributed to the transitions from the ground state to the higher states of rare-earth ions are assigned. As calculated from the absorption spectrum, the absorption cross section (σ_{abs}) can be obtained[13]

$$\sigma_{abs} = \frac{2.303 \times O_D}{Nl}, \tag{2}$$

where O_D represents the optical density, N represents the concentration of rare-earth ions, and l is the thickness of the sample. The peak absorption cross section of the $Er^{3+}: {}^4I_{15/2} \rightarrow {}^4I_{9/2}$ and $Nd^{3+}: {}^4I_{9/2} \rightarrow {}^4F_{5/2}, {}^2H_{9/2}$ transitions are 1.26×10^{-21} cm^2 and 29.88×10^{-21} cm^2, respectively. This result can indicate the probability of energy transfer between $Er^{3+}: {}^4I_{9/2}$ and $Nd^{3+}: {}^4F_{5/2}, {}^2H_{9/2}$ levels of the co-doped glass when pumped by 808 nm laser diode.

According to the absorption spectrum, the Judd – Ofelt (J-O) parameters are obtained with the exception of the transition $Er^{3+}: {}^4I_{15/2} \rightarrow {}^4I_{13/2}$ using the least-squares fitting method[14-15]. Intensity parameters values Ω_t of Er^{3+} ion in bismuth germanate glasses and various other glasses hosts are listed in Table 1 (the root mean square deviation is 0.44×10^{-6}). As it's known, the parameter Ω_2 is strongly affected by the covalent bonding and the local environmental asymmetry around RE ions. Meanwhile, Ω_4 and Ω_6 are sensitive to the viscosity and rigidity of glass. As shown in Table 1, the parameter Ω_2 of the used glass is higher than that of FP glasses, but lower than that of chalcohalide and germanate glasses. It is known that oxygen ions (3.44) are more polarizable than fluoride ions (3.98), due to its higher electronegativity[8]. The sulfur ions (2.58) have the lowest electronegativity in comparison with oxygen ions. As a result, the covalence of oxygen glasses is stronger than that of fluoride glasses, but weaker than that of chalcohalide glasses. Moreover, the covalent degree of Ge-O bond is stronger than that of Bi-O bond. In addition, the radiative transition probability and branching ratio of the $Er^{3+}: {}^4I_{11/2} \rightarrow {}^4I_{13/2}$ transition in samples is listed in Table 1. The radiative transition probability (58.46 s^{-1}) in bismuth germanate glasses is much larger than other glasses, Thus, the studied Er^{3+}/Nd^{3+} doped bismuth germanate glasses may be a good alternative material for the mid-infrared laser.

Table 1 J-O parameters, spontaneous emission probability and branching ratio of $Er^{3+}: {}^4I_{11/2} \rightarrow {}^4I_{13/2}$ transition in various glasses

Glasses	Ω_2 ($\times10^{-20}$ cm^2)	Ω_4 ($\times10^{-20}$ cm^2)	Ω_6 ($\times10^{-20}$ cm^2)	Ω_4/Ω_6	A_{rad} (s^{-1})	β /%
FP[8]	3.59	1.34	0.93	1.44	24.11	17.68
FP[9]	3.27	0.75	2.33	0.32	31.23	15
Chalcohalide[7]	6.37	1.41	0.73	1.93	48.40	16.6
Germanate[3]	6.6	1.5	0.9	1.67	47.8	14.4
Bismuth germanate	4.91	1.54	0.90	1.71	58.46	17.26

3.2 Emission spectra and emission cross section

Figure 2 shows the emission spectra around 2.7 μm and 1.5 μm of Er^{3+} single doped and Er^{3+}/Nd^{3+} co-doped samples. The emission intensity around 2.7 μm assigned to the $Er^{3+}:{}^4I_{11/2}\rightarrow{}^4I_{13/2}$ transition is strongly enhanced, whereas the 1.5 μm emission of Er^{3+} is greatly suppressed by the co-doping Nd^{3+}. The lifetime of $Er^{3+}:{}^4I_{13/2}$ level drasticly decreases from 8.87 ms to 1.33 ms by Nd^{3+} co-doping. These behaviors are mainly attributed to the high energy transfer efficiency between Nd^{3+} and Er^{3+} ions, which increases the population of the excited $Er^{3+}:{}^4I_{11/2}$ level and reduces the population remained in $Er^{3+}:{}^4I_{13/2}$ level. Additionally, the Er^{3+} single doped sample with the higher OH^- content should possess the lower emssion intensity of $Er^{3+}:{}^4I_{13/2}$ level in comparison with the codoped sample. However, the concentrations of hydroxyl group in these two samples are relatively low so that the intensity of $Er^{3+}:{}^4I_{13/2}$ level is under significant influence of the codoped Nd^{3+} ions. It can be deduced that the Er^{3+} ions can be efficiently sensitized by the co-doped Nd^{3+} ions to realize the 2.7 μm emission in bismuth germanate glasses. Energy transfer mechanism between Nd^{3+} and Er^{3+} will be discussed below. In addition, the optimum concentration ($Er^{3+}:Nd^{3+}=1:1$) is found from a series of samples with different ratios because this ratio exhibits the strongest mid-infrared emission in bismuthate glasses.

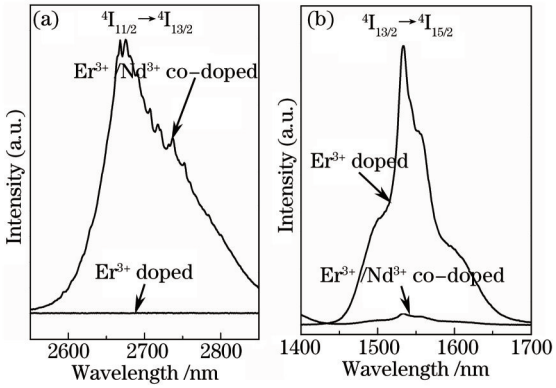

Fig.2 (a) 2.7 μm and (b) 1.5 μm emission spectra of Er^{3+}- and Er^{3+}/Nd^{3+}-doped bismuth germanate glass under 808 nm excitation

The 2.7 μm emission cross section (σ_{em}) can be calculated based on Fuchtbauer-Ladenburg theory and Fig.2 using the following equation[16-17]

$$\sigma_{em}=\frac{\lambda^4 A_{rad}}{8\pi cn^2}\times\frac{\lambda I(\lambda)}{\int\lambda I(\lambda)\mathrm{d}\lambda}, \tag{3}$$

where λ represents the wavelength, A_{rad} represents the spontaneous transition probability corresonding to $Er^{3+}:{}^4I_{11/2}$ to $Er^{3+}:{}^4I_{13/2}$ transition, c and n are the light speed in vacumn and the glass refractive index respectively, $I(\lambda)$ represents the emssion magnitude. As a result, the peak of σ_{em} in the Er^{3+}/Nd^{3+}-doped samples is up to 8.34×10^{-21} cm^2. It is found that the emission cross section in bismuth germanate glasses is higher than that of chalcohalide (6.6×10^{-21} cm^2)[7] and germanate glasses (7.02×10^{-21} cm^2)[3].

3.3 Energy transfer microparameters and efficiency

The energy level diagram as well as the involved energy transfer channels between Er^{3+} and Nd^{3+} ions is shown in Fig.3. Firstly, Er^{3+} and Nd^{3+} ions are pumped to $Er^{3+}:{}^4I_{9/2}$ and $Nd^{3+}:{}^4F_{5/2}$, ${}^2H_{9/2}$ level from the respective ground state under 808 nm excitation. On one hand $Er^{3+}:{}^4I_{9/2}$ de-excites nonradiatively to $Er^{3+}:{}^4I_{11/2}$, part of the population in $Er^{3+}:{}^4I_{11/2}$ level relaxes to the next lower $Er^{3+}:{}^4I_{13/2}$ level, with the emission being centered around 2.7 μm. Subsequently, the radiative relaxation of $Er^{3+}:{}^4I_{13/2}$ to the ground state $Er^{3+}:{}^4I_{15/2}$ yield 1.5 μm emission. On the other hand, $Nd^{3+}:{}^4F_{5/2}$, ${}^2H_{9/2}$ de-excites nonradiatively to $Nd^{3+}:{}^4F_{3/2}$ level , then it relaxes to $Nd^{3+}:{}^4I_{11/2}$ yielding 1.06 μm emission. Finally, Er^{3+} and Nd^{3+} ions relax to the ground state $Er^{3+}:{}^4I_{15/2}$ and $Nd^{3+}:{}^4I_{9/2}$ level. It is also worth noting several shortcuts between $Er^{3+}$and Nd^{3+} ions in Fig.3:

$$^4F_{5/2},{}^2H_{9/2}(Nd^{3+})+{}^4I_{15/2}(Er^{3+})\rightarrow{}^4I_{9/2}(Nd^{3+})+{}^4I_{9/2}(Er^{3+}), \tag{4}$$

$$^4F_{3/2}(Nd^{3+})+{}^4I_{15/2}(Er^{3+})\rightarrow{}^4I_{9/2}(Nd^{3+})+{}^4I_{11/2}(Er^{3+}), \tag{5}$$

$$^4I_{9/2}(Nd^{3+}) + {}^4I_{13/2}(Er^{3+}) \rightarrow {}^4I_{15/2}(Nd^{3+}) + {}^4I_{15/2}(Er^{3+}), \tag{6}$$

equation (4) is a quasiresonant energy transfer process, however, equation (5) is a nonresonant process. So several phonons are needed to assist the equation (5) process. The two processes are beneficial to the improvement of ions in $Er^{3+}:{}^4I_{11/2}$ level. The depopulation of $Er^{3+}:{}^4I_{13/2}$ decreases the 1.5 μm emission via equation (6) process. As a result, the three shortcuts significantly enhance the emission at 2.7 μm and decrease the emission at 1.5 μm.

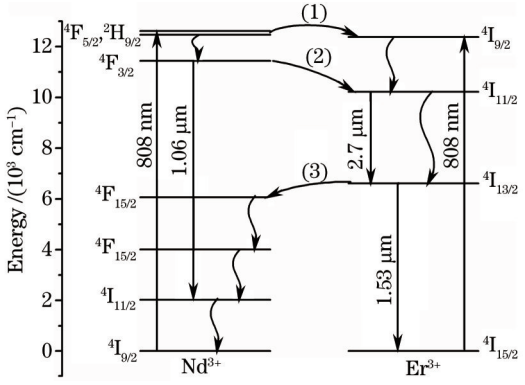

Fig.3 Energy transfer sketch of Er^{3+}/Nd^{3+}-doped bismuth germanate glass when pumped at 808 nm

According to the theory of energy transfer mechanism among ions in solid driven by Dexter[18] and Förster[19], the energy transfer coefficient (cm^6/s) in ET2 process between Nd^{3+} (donor, D) and Er^{3+} (acceptor, A) are estimated as

$$C_{DA} = \frac{6cg^D_{low}}{(2\pi)^4 n^2 g^D_{up}} \sum_{m=0}^{N} \exp[-2\bar{n}+1] S_0 \frac{S_0^m}{m!} (\bar{n}+1)^m \times \int \sigma^D_{em}(\lambda_m^+) \sigma^A_{abs}(\lambda) d\lambda , \tag{7}$$

$$C_{AD} = \frac{6cg^A_{low}}{(2\pi)^4 n^2 g^A_{up}} \sum_{k=0}^{N} \exp(-2\bar{n}S_0) \frac{S_0^k}{k!} (\bar{n})^k \times \int \sigma^A_{em}(\lambda_k^-) \sigma^D_{abs}(\lambda) d\lambda , \tag{8}$$

Then, the energy transfer probability rate is estimated in the following equation[20]

$$W_{DA}(R) = \frac{C_{DA}}{R^6} , \tag{9}$$

$$W_{AD}(R) = \frac{C_{AD}}{R^6} , \tag{10}$$

where R is the separate distance between the donor and the acceptor.

The energy transfer microparameters of ET2 process in the co-doped sample are calculated through the equations (7)~(10) and listed in Table 2. The results indicate that the direct-transfer needs the assistance of 2 (95%), 3(5%) phonons emission in Nd^{3+} site, while the respective back-transfer needs the assistance of 2 (31%), 3 (55%), 4 (14%) phonons absorption. Therefore, the direct energy transfer $Nd^{3+}:{}^4F_{3/2} \rightarrow Er^{3+}:{}^4I_{11/2}$ is more effective than the back energy transfer $Er^{3+}:{}^4I_{11/2} \rightarrow Nd^{3+}:{}^4F_{3/2}$ (nearly 8 times higher.). In addition, the obtained energy transfer probability rate of $Nd^{3+}:{}^4F_{3/2} \rightarrow Er^{3+}:{}^4I_{11/2}$ is 2.48×10^3 s^{-1}, which is larger than that in FP glasses (2.01×10^3 s^{-1}) [8] indicates that energy transfer from Nd^{3+} to Er^{3+} in bismuth germanate glasses is more effcient than that in FP glasses.

Table 2 Calculated microparameters C_{DD} for $Yb^{3+} \rightarrow Yb^{3+}$ energy migration and C_{DA} for $Yb^{3+} \rightarrow Tm^{3+}$ energy transfer. The number (#) of phonons needed to assist the energy transfer as well as the percentage of each phonon participation (%) in the process.

Energy transfer	N(# of phonons) (phonon-assist, %)	C_{DA} (10^{-40}cm^6/s)	W_{DA} (s^{-1})
$Nd^{3+}:{}^4F_{3/2} \rightarrow Er^{3+}:{}^4I_{11/2}$	2, 3, 4 (95, 5, 0%)	6.90	2483
$Er^{3+}:{}^4I_{11/2} \rightarrow Nd^{3+}:{}^4F_{3/2}$	2, 3, 4 (31, 55, 14%)	0.93	335

In the Er^{3+}/Nd^{3+} co-doped glass, the energy transfer efficiency from $Er^{3+}:{}^4I_{13/2}$ to $Nd^{3+}:{}^4I_{15/2}$ is given by the following formula[18]

$$\eta = 1 - \tau_c/\tau_s , \tag{11}$$

where τ_c and τ_s are the lifetimes of $Er^{3+}:{}^4I_{13/2}$ level monitored at 1.5 μm, in Er^{3+}/Nd^{3+} co-doped and Er^{3+} single doped glasses respectively. Figure 4 shows the decay curves at 1.5 μm fluorescence in two samples upon 808 nm excitation. The

calculated energy transfer efficiency is up to 85%. The high energy transfer of Er^{3+}:$^4I_{13/2}$→Nd^{3+}:$^4I_{15/2}$ level decreases rapidly the ions in Er^{3+}:$^4I_{13/2}$ level. In a word, under 808 nm excitation, the introduced Nd^{3+} ion is highly efficient for the improvement of 2.7 μm emission in bismuth germanate glasses.

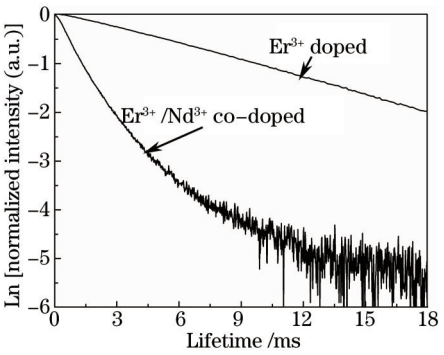

Fig.4 Fluorescence decay curves of Er^{3+}:$^4I_{13/2}$ level in Er^{3+}-doped bismuth germanate glass with and without Nd^{3+} pumped by 808 nm

4 Conclusions

The enhanced emission around 2.7 μm in the Er^{3+}/Nd^{3+}-doped bismuth germanate glasses can be achieved upon 808 nm excitation. The spectroscopy properties of Er^{3+}/Nd^{3+}-doped glasses are analyzed in terms of the J-O intensity parameters and radiative transition probability. The peak emission cross section for Er^{3+}:$^4I_{11/2}$→$^4I_{13/2}$ transition is equal to 8.34×10^{-21} cm^2. Large energy transfer probability rate of Nd^{3+}:$^4F_{3/2}$→Er^{3+}:$^4I_{11/2}$ (2.48×10^3 s^{-1}) and high energy transfer efficiency (85%) between Er^{3+}:$^4I_{13/2}$ and Nd^{3+}:$^4I_{15/2}$ levels indicate the efficient sensitization of Nd^{3+} to 2.7 μm emission of Er^{3+} ions. The Er^{3+}/Nd^{3+}-doped bismuth germanate glasses is a potential material that can be utilized in mid-IR laser applications.

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