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An investigation of 3.5 μm emission in Er^{3+}-doped fluorozirconate glasses under 638 nm laser excitation

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Abstract

Intense 3.5 μm mid-infrared emission has been achieved in Er^{3+}-doped ZBYA glasses, which is ascribed to the Er^{3+}: $^4F_{9/2}\rightarrow{}^4I_{9/2}$ transition. Based on the absorption spectrum of Er^{3+} ions, a 638 nm laser was utilized to directly pump the upper level (Er^{3+}: $^4F_{9/2}$) to achieve 3.5 μm emission with enhanced quantum efficiency. Spectroscopic parameters were predicted by Judd-Ofelt theory. The maximum emission cross-section of the Er^{3+}-doped ZBYA glass was estimated to be 5.5×10^{-22} cm^2 at 3496 nm. Additionally, the fluorescence spectra and energy level lifetimes of ZBYA glass samples with different Er^{3+} ions doping concentrations were also measured. The theoretical and experimental results confirm the potential of Er^{3+}-doped ZBYA glasses for use in the development of 3.5 μm mid-infrared fiber lasers.

Keywords: Mid-infrared; ZBYA glass; Er^{3+}-doped; quantum efficiency

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1. Introduction

Mid-infrared (MIR) laser sources in the wavelength range around 3-5 μm play an essential role in applications areas such as gas spectroscopy, atmospheric communications, biomedical and material processing fields [1-3]. In the past few decades, fiber lasers based on Er^{3+} ions as activators have attracted intensive study as MIR sources [4-6]. Lasing in Er^{3+}-doped ZBLAN fiber has been widely investigated, generating MIR emission at λ~2.8 μm through the Er^{3+}: $^4I_{11/2}\rightarrow{}^4I_{13/2}$ transition using a 980 nm laser as a pump source [7]. Up to now, the highest power achieved for an MIR fiber laser is 70 W at λ~2.8 μm [8]. In more recent years, laser emission at λ~3.5 μm, corresponding to the transition Er^{3+}: $^4F_{9/2}\rightarrow{}^4I_{9/2}$, has attracted a lot of interest, given that such lasers can

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*Corresponding author: pengfei.wang@tudublin.ie be used for in variety of application areas such as polymer processing and molecular spectroscopy [9, 10]. Early in 1992, H. Többen *et al.* demonstrated 3.5 μm laser emission from an Er^{3+}-doped ZBLAN fiber at room temperature pumped by a dicyanomethylene (DCM) dye laser at 655 nm [11]. In such a laser, Er^{3+} ions will be excited directly to the Er^{3+}: $^4F_{9/2}$ level by absorbing 655 nm energy and will then relax to a lower level resulting in 3.5 μm emission. However, the disadvantages of pumping at 655 nm were that the threshold power was high at 996 mW and the slope efficiency was poor at 2.8% due to relatively high multiphonon rate. In 2016, Ori Henderson-Sapir *et al.* reported a dual-wavelength pumping (DWP) scheme to implement a Er^{3+} ion 3.5 μm laser [12]. By utilizing both 985 nm and 1973 nm wavelengths as pump sources, particles in the ground state were excited to the upper energy level for 3.5 μm emission. In 2022, Maxime Lemieux-Tanguay *et al.* proposed a DWP all-fiber continuous-wave (CW) laser operating at 3.55 μm using Er: ZBLAN fiber which reached an output power of 14.9 W. This laser involved splicing silica fiber to the ZBLAN fiber and using two fiber Bragg gratings (FBGs) as laser reflectors [13]. In the same year, Qin Zhipeng *et al.* investigated a 658 nm diode-pumped CW and mode-locked fiber laser at 3.5 μm. Using a 7.0 mol.% Er: ZBLAN gain fiber, a CW output power of 203 mW was experimentally obtained at 3462 nm [14]. While DWP schemes can deliver lasers with high output power, single wavelength pumping schemes are more desirable since they not only simplify the pump source but also reduce the complexity of coupling. This in turn can lead to cost reductions which will promote the application of 3.5 μm MIR fiber lasers in a wide range of applications. Er: ZBLAN fiber as gain medium has yielded promising results, but two major drawbacks exist: a low glass transition temperature (T_g) and poor chemical durability. Overall then progress in the MIR fiber laser area is restricted by the lack of high-performance gain medium.

ZrF_4-BaF_2-YF_3-AlF_3 (ZBYA) fluorozirconate glass is considered to be a promising gain medium for MIR lasing emission. It has known merits that include a low phonon energy (571 cm^{-1}), a higher chemical stability and glass transition temperature than ZBLAN glass, a higher damage threshold tolerance and a more stable composition than fluoroindate glass [15, 16]. Recently, our group reported a 2.9 μm MIR laser from an in-house fabricated Ho^{3+}/Pr^{3+} co-doped ZBYA glass fiber where the maximum unsaturated output power reached 2.16 W [17]. This indicates that ZBYA glass is a useful gain medium for a MIR laser. In this paper, we prepared Er^{3+}-doped ZBYA glasses using conventional melt-quenching method in a glove box. Under 638 nm laser diode (LD) excitation, intense 3.5 μm emission was observed in Er: ZBYA glasses and the fluorescence properties for lasing have been studied.

2. Experiment

The Er^{3+}-doped ZBYA glasses were prepared through the conventional melt-quenching method in a glove box to avoid contamination by water. First, high-purity ZrF_4 (99.99%), BaF_2 (99.99%), YF_3 (99.99%), AlF_3 (99.9%) and ErF_3 (99.99%) were employed as raw materials. These powder samples were weighed and mixed evenly in the ratio: 50 ZrF_4 - 33 BaF_2 - (10-x) YF_3 -7 AlF_3 - x ErF_3 (x=0.1, 0.2, 0.5, 1, 2, 3, 4, 5, 7, 9) (mol%). Batches of 15 g were thoroughly mixed and then melted in a covered platinum crucible at 850 °C for 2 h. After that, the melt was cast into a preheated brass plate (320 °C) for 3 h to relieve stress. The bulk samples were cut into blocks and optically polished with a thickness of 2 mm for subsequent measurements.

The absorption spectrum of the glass sample was measured using a Perkin-Elemer Lambda 900 UV spectrophotometer over the wavelength range of 200-1800 nm. Under the excitation of an in-house fabricated 638 nm LD, fluorescence emission spectra were detected by a Zolix Omni-λ300i fluorescence spectrometer with an InGaAs detector or liquid nitrogen cooled InSb detector, which were used to measure the near and mid-infrared emission spectra, respectively. The emission spectra of Er: ZBYA glasses were measured when pumped by a 638 nm laser diode at 1.0 W with a beam waist of 2 mm. Fluorescence decay curves were detected and recorded using a fluorescence spectrometer (Techcomp FLS1000), with an optical parametric oscillator (Horizon II OPO) used as the pump source. The parametric oscillator wavelength was tuned to λ~630 nm, with a pulse width of 6 μs and repetition rate was 10 Hz. All measurements were performed at room temperature.

3. Results

The absorption spectrum of 1.0 mol% Er^{3+}-doped ZBYA glass is shown in Fig. 1, the wavelength range is 200 nm-1800 nm with a test step size of 1 nm. The absorption peaks of 378 nm, 520 nm, 640 nm, 800 nm, 980 nm and 1530 nm correspond to the transitions from the ground state $^4I_{15/2}$ to the excited state energy levels of $^4G_{11/2}$, $^2H_{11/2}$, $^4F_{9/2}$, $^4I_{9/2}$, $^4I_{11/2}$, and $^4I_{13/2}$, respectively, which have been marked in Fig. 1.

Since the high efficiency 3.5 μm fluorescence emission of Er^{3+} ions is main objective in this work, the upper level $^4F_{9/2}$ of the 3.5 μm emission is chosen to be directly pumped to improve the luminescence quantum efficiency. Based on the absorption peak of the Er^{3+}: $^4F_{9/2}$ level in the 640 nm band, an in-house fabricated 638 nm LD was used as the pump source. The energy level diagram of Er^{3+} ions under the excitation of a 638 nm pumping is shown in the inset of Fig. 1.

As shown in the inset Fig. 1, when the samples are excited by the 638 nm laser, the ions on the $^4I_{15/2}$ ground level are pumped to the $^4F_{9/2}$ level through ground state absorption (GSA). Then a portion of the ions on the $^4F_{9/2}$ level transition back to the $^4I_{9/2}$ level, producing fluorescence emission in the mid-infrared of 3.5 μm band and further returning to the $^4I_{15/2}$ level with 800 nm radiation; while further portion of the ions relax to $^4I_{13/2}$ level with 1150 nm emission. The ions that occupy the $^4I_{9/2}$ level decay non-radiatively to the next level $^4I_{11/2}$ as well as undergoing a further rapid decay to the $^4I_{13/2}$ level, which in turn produces luminescence in the 2.7 μm band. Another portion of the ions in the $^4I_{11/2}$ level relax to the ground level via radiative processes, yielding 980 nm emission. Furthermore, most of the Er^{3+} ions in the $^4I_{13/2}$ level radiatively transition to the ground energy level and gave rise to 1550 nm fluorescence.

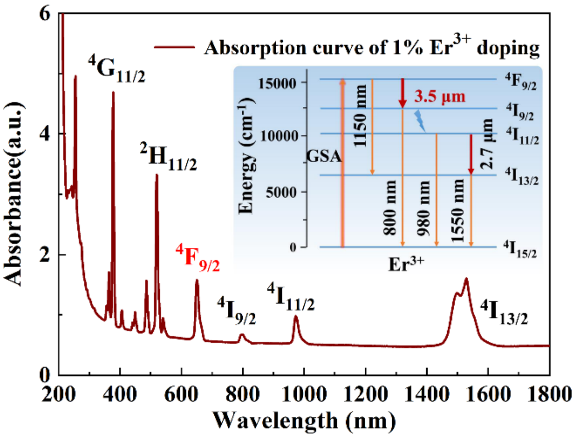

Fig. 1. Absorption spectrum of 1.0 mol% Er^{3+} doped ZBYA glass; Inset: Er^{3+} ion level diagram under λ~638 nm laser excitation.

Based on the measured absorption spectrum for Er^{3+} ions, the Judd-Ofelt (J-O) intensity parameters Ω_t (t=2, 4, 6) for Er^{3+}-doped ZBYA glass were calculated as 2.373×10^{-20} cm^2, 0.288×10^{-20} cm^2 and 1.977×10^{-20} cm^2, respectively. The J-O intensity parameters Ω_t (t=2, 4, 6) for Er^{3+} ions in several glass host matrices are listed in Table 1. In general, Ω_2 provides information on the local environment for rare earth ions sites and the point symmetry of the environment around the Er^{3+} ions. Ω_6 reflects the photo-alkalinity of the matrix glass, a larger Ω_6 is associated with lower photo-alkalinity. Therefore, J-O analysis shows that the prepared Er: ZBYA glass has a stronger covalency and lower photo-alkalinity.

Table 1 Calculated J-O intensity parameters Ωt (t=2, 4, 6) (10^{-20} cm^2) of Er^{3+} ion in various glass hosts

Material	Ω_2	Ω_4	Ω_6
Silicate [18]	4.23	1.04	0.61
ZBLAN [18]	2.91	1.27	1.11
Fluoroaluminate [19]	2.021	1.194	1.239
Fluoroindate [20]	2.8815	2.2337	1.267
ZBYA [This work]	2.373	0.288	1.977

Table 2 J-O calculated parameters for selected transitions of Er^{3+} ion in ZBYA glass

Transition	Wavelength (nm)	A_{rad} (s^{-1})	β (%)	τ_{rad} (ms)
$^4F_{9/2} \rightarrow {}^4I_{9/2}$	3500	44.78	2.4	0.54
$\rightarrow {}^4I_{11/2}$	1920	140.6	7.55	
$\rightarrow {}^4I_{13/2}$	1150	412.5	22.14	
$\rightarrow {}^4I_{15/2}$	660	1265.36	67.91	
$^4I_{9/2} \rightarrow {}^4I_{11/2}$	4500	2.04	2.6	12.78
$\rightarrow {}^4I_{13/2}$	1700	14.28	18.25	
$\rightarrow {}^4I_{15/2}$	800	61.94	79.15	
$^4I_{11/2} \rightarrow {}^4I_{13/2}$	2800	44.03	11.27	2.56
$\rightarrow {}^4I_{15/2}$	980	346.55	88.73	
$^4I_{13/2} \rightarrow {}^4I_{15/2}$	1500	183.19	100	5.46

Based on the J-O parameters, Table 2 summarizes the radiative parameters of ZBYA glass samples for different transfer processes, including the emission wavelength, spontaneous emission transition probability (A_{rad}), fluorescence branching ratio (β) and theoretical radiative lifetime (τ_{rad}). The spontaneous emission transition probability (A_{rad}), fluorescence branching ratio (β) and theoretical radiative lifetime (τ_{rad}). The spontaneous emission transition probability of the transition Er^{3+}: $^4F_{9/2} \rightarrow {}^4I_{9/2}$ is calculated as 44.78 s^{-1}, which is higher than that of ZBLAN glass (~26.3 s^{-1}) [21], indicating that ZBYA glass has the potential to be an efficient candidate material for a fiber laser operating at λ~3.5 μm.

To examine the characteristics of λ~3.5 μm emission for potential laser applications, the emission cross-section (σ_{emi}) and absorption cross-section (σ_{abs}) of $^4F_{9/2} \rightarrow {}^4I_{9/2}$ transition were calculated by Fuchtbauer-Ladenburg (FL) theory [22]:

$$\sigma_{\mathrm{emi}}(\lambda)=\frac{A_{rad}}{8\pi c n^2}\times\frac{\lambda^5 I(\lambda)}{\int \lambda I(\lambda)\mathrm{d}\lambda} \qquad (1)$$

where λ is the center wavelength of the fluorescence spectrum, *I(λ)* is the emission intensity, A_{rad} is spontaneous transition probability, c is the speed of the light, and n is the refractive index of the glass.

$$\sigma_{\mathrm{abs}}(\lambda)=\sigma_{\mathrm{emi}}\frac{Z_U}{Z_L}\exp\left[\frac{\mathrm{hc}/\lambda-E_{ZL}}{\mathrm{k_B T}}\right] \qquad (2)$$

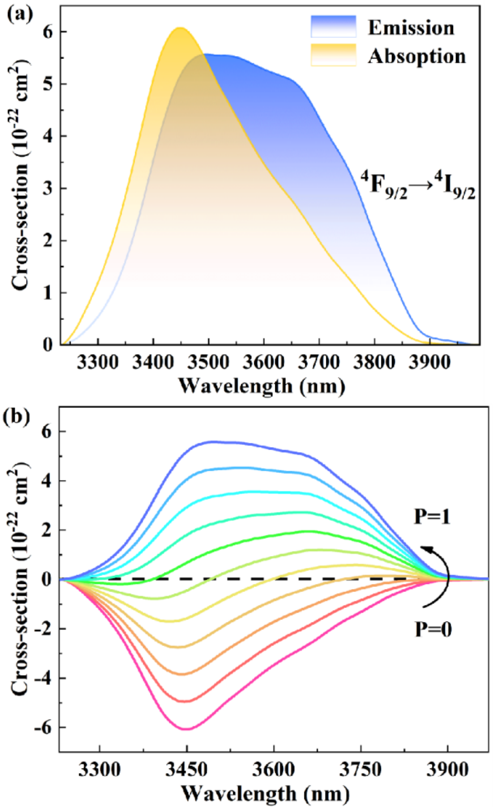

Fig. 2. (a) λ~3.5 μm emission and absorption cross-section and (b) gain cross-section.

where Z_L and Z_U are the partition functions of lower and upper levels, respectively. k_B is the Boltzmann constant, T represents the room temperature, and E_{ZL} represents the zero-line energy, which has the physical meaning of the free energy required to excite an ion from a low to a high energy level at temperature T. The gain coefficient $G(\lambda)$ can be calculated by the equation:

$$G(\lambda) = P\sigma_{emi}(\lambda) - (1 - P)\sigma_{abs}(\lambda) \tag{3}$$

where P is the ratio of the population of the upper energy levels to the population of total energy levels. The calculated results are shown in Fig. 2 (a), the maximum emission cross section of 5.5×10^{-22} cm^2 is obtained at 3496 nm. The gain coefficients for various values of P ranging from 0 to 1 were also calculated for the $^4F_{9/2}\rightarrow{}^4I_{9/2}$ transition in ZBYA glass, the gain cross-section is shown in Fig. 2(b). It is evident that a positive gain is obtained when $P>0.3$, which implies that a low pumping threshold can be achieved for laser operation based on the $^4F_{9/2}\rightarrow{}^4I_{9/2}$ transition [23].

To demonstrate the fluorescence characteristic of ZBYA glasses in the 3.5 μm band with different Er^{3+} doping concentrations, mid-infrared emission spectra in the wavelength region 3200-4000 nm were measured under the excitation of a 638 nm LD, as depicted in Fig. 3. The luminescence intensity of the emission band is centered at 3496 nm, originating from the Er^{3+}: $^4F_{9/2}\rightarrow{}^4I_{9/2}$ transition. It shows a rising intensity with increases in the Er^{3+} ion doping concentration, furthermore.

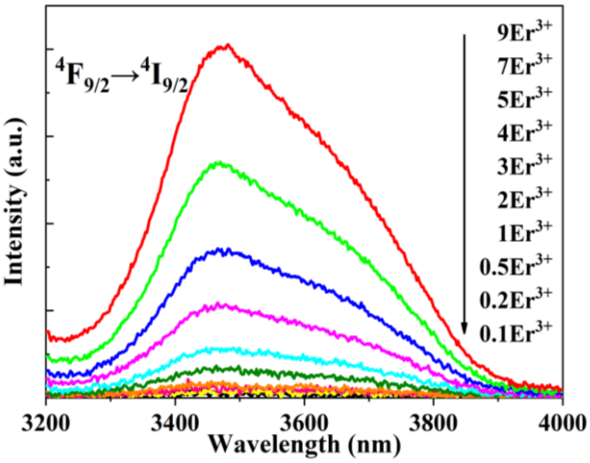

Fig. 3. λ~3.5 μm mid-infrared luminescence spectra for different doping concentrations of Er^{3+} in ZBYA glasses under λ~638 nm laser excitation.

In order to further explore the luminescence properties of Er^{3+}-doped ZBYA glasses, the fluorescence spectra at λ~980 nm, λ~1150 nm, λ~1550 nm and λ~2.7 μm corresponding to the transition of $^4I_{9/2}\rightarrow{}^4I_{15/2}$, $^4F_{9/2}\rightarrow{}^4I_{13/2}$, $^4I_{13/2}\rightarrow{}^4I_{15/2}$ and $^4I_{11/2}\rightarrow{}^4I_{13/2}$, respectively, were recorded and are shown in Fig. 4. In a similar manner to the 3.5 μm case, luminescence intensity in each band increases with an increase in the doping concentration of Er^{3+} ions.

For the lifetimes testing, the glass samples were excited at 630 nm in the experiment, the fluorescence decay curves at 660 nm, 980 nm and 1550 nm bands were monitored. As shown in Fig. 5 (a)-(c), the lifetimes of $^4F_{9/2}$, $^4I_{11/2}$ and $^4I_{13/2}$ levels in the 1 mol% Er^{3+}-doped glass samples are 0.121 ms, 7.02 ms and 11.29 ms, respectively. Fig. 5 (d) shows the measured

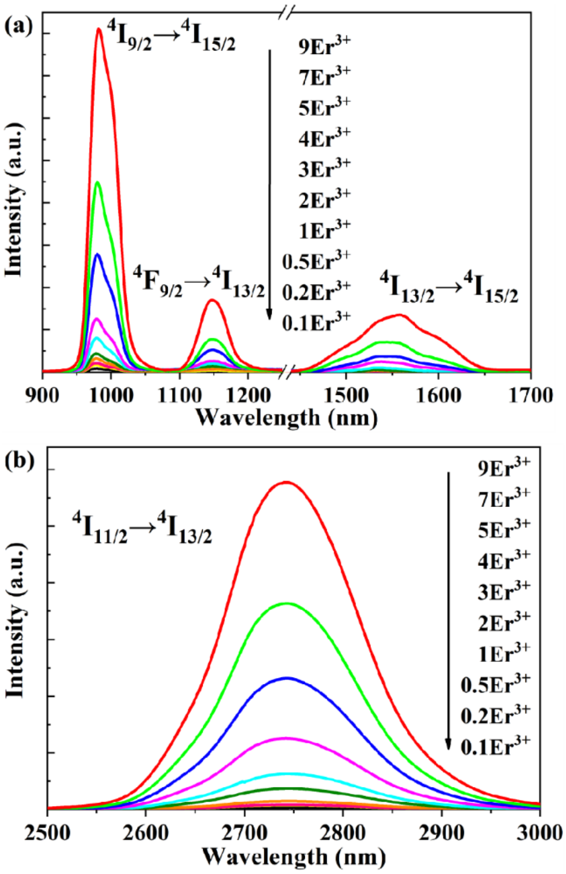

Fig. 4. (a) Near-infrared and (b) λ~2.7 μm mid-infrared luminescence spectra of different doping concentration Er^{3+}-doped ZBYA glasses under λ~638 nm laser excitation.

lifetimes for $^4I_{13/2}$, $^4I_{11/2}$ and $^4F_{9/2}$ levels as a function of Er^{3+} doping concentration. Based on the results, it can be seen that the lifetimes of $^4I_{11/2}$ and $^4F_{9/2}$ levels are almost constant with an increase in the Er^{3+} ion doping concentration, while the lifetimes for the $^4I_{13/2}$ level show a modest upward trend with the increase of Er^{3+} ion doping concentration, reaching a maximum of 13.78 ms. The lifetime of $^4I_{9/2}$ level is too short to be detected as a result of the equipment detection accuracy, but it is reported in the literature to be about 8 μs [24].

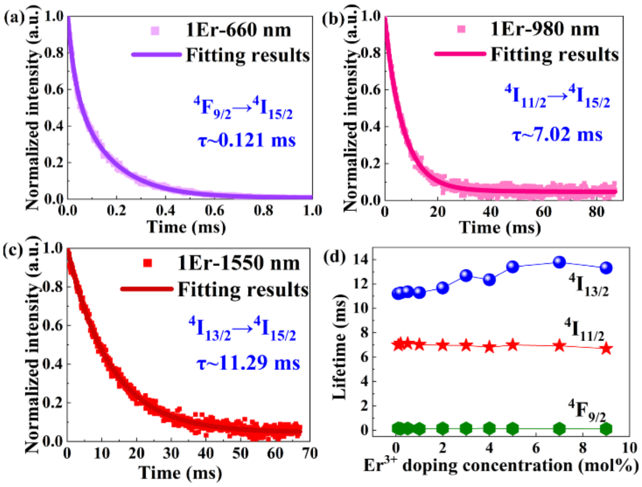

Fig. 5. Energy level lifetime of (a) 660 nm: $^4F_{9/2}\rightarrow{}^4I_{15/2}$, (b) 980 nm: $^4I_{11/2}\rightarrow{}^4I_{15/2}$ and (c) 1550 nm: $^4I_{13/2}\rightarrow{}^4I_{15/2}$ in the 1 mol% Er^{3+}-doped sample; (d) Energy level lifetimes of Er^{3+}: $^4I_{13/2}$, $^4I_{11/2}$, $^4F_{9/2}$ levels as a function of Er^{3+} doping concentration under λ~630 nm laser.

The quantum efficiency η is usually used to assess the ability of lasing for glass gain materials. In practical experiments, the quantum efficiency is connected with the maximum phonon energy of the substrate glass. η can be calculated by following equation [25]:

$$\eta = \frac{\tau_{mea}}{\tau_{rad}} \times 100\% \qquad (4)$$

where τ_{mea} is the spontaneous radiative lifetime calculated from J-O parameters and τ_{rad} is the measured fluorescence lifetime. The quantum efficiency of Er^{3+}-doped ZBYA glass for the 3.5 μm band is calculated as 27.78%, the result is higher than fluoroindate glass (25.0%) [20], which confirms the considerable potential application of Er: ZBYA glass as a gain medium for λ~3.5 μm fiber lasers.

4. Conclusion

In conclusion, intense 3.5 μm MIR luminescence emission has been demonstrated in Er^{3+}-doped ZBYA glasses for the first time. The gap between the upper and lower energy levels is small for the 3.5 μm band transition, which results in an inefficient lasing radiation due to high quantum defect in the matrix with a high phonon energy. The low phonon energy of the ZBYA glass material in this experiment can reduce the non-radiative relaxation process of multiple phonons in the matrix, and the $^4F_{9/2}$ upper energy level is directly excited by a 638 nm laser, which in turn can improve the fluorescence quantum efficiency of the 3.5 μm band. The absorption, emission and gain cross-sections were determined, yielding a maximum emission cross-section of 5.5×10^{-22} cm^2 at 3496 nm. The quantum efficiency for the 3.5 μm band is calculated as 27.78%. As a consequence of these favorable spectroscopic characteristics, it is possible to assert that Er^{3+}-doped ZBYA glasses are strong candidates as host materials for 3.5 μm MIR fiber lasers.

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