Concentration dependent enhanced luminescence and its quenching effect in erbium incorporated heavy metal oxide—borate glasses

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Abstract

Erbium incorporated zinc-lead-bismuth-borate glasses are prepared through melt-quenching technique and their concentrated dependent structural, thermal and spectroscopic properties are evaluated. Prepared glasses are thermally stable and amorphous nature and the density and refractive indices of the prepared glasses increases with Er₂O₃ concentration. UV–vis–NIR spectra exhibits multiple transitions accompanied by hyper sensitive transition of ⁴I_{15/2} → ²H_{11/2}. Theoretical Judd-Olfet calculations were performed and correlated with experimental results. JO and bonding parameters reveal the covalence nature of the prepared glasses and radiative probabilities shows highest branching ratio for ⁴I_{15/2} → ²H_{11/2} transition. Luminescence results exhibit luminescence enhancement up to 1 mol% of erbium and thereafter luminescence quenches due to the existence of cross-relaxation channels. Using McCumber’s theory, absorption and emission cross-section values are estimated and are in agreement with each other and as well as with the experimental stimulated cross-sectional values. About 40% population inversion is achieved for the prepared glasses. Therefore, the investigated results suggest that, the prepared glasses are suitable candidates for amplifier applications.

1. Introduction

In recent years, erbium doped fiber amplifiers are drawing much demand as a potential candidate for various efficient devices for technological applications such as optical amplifiers, fiber networks and photonic devices [1]. The transition spectra of Er ion consist of very strong intensities and sharp spectral characteristics in ⁴f transitions [2]. In general, the optical transitions of rare earth ion depend on the type of host material. Numerous reports are available on the utilization of the different host to achieve high optical parameters. Selecting suitable host is a vital task to attain minimum loss, high luminescence properties [3]. It is reported that, borate is cost effective, structurally deformable former and it has high phonon energy. In contrast, heavy metal oxides are having high polarizability capacity and low phonon energy. In addition, heavy metal oxides are suitable candidates for enhancing optical properties due to their larger linear refractive index, high nonlinear response, smaller cut off phonon energy (500–700 cm⁻¹) and good physio-chemical properties [4, 5]. When borate is replaced by heavy metal oxides, the non-radiative losses can be reduced and intern the luminescence properties can be enhanced [6].

Apart, the amplification of Erbium doped fiber amplifiers (EDFA) at 1550 nm is reported [7]. However, erbium and other rare-earth ions incorporated matrix exhibits narrow gain bandwidths (about 100 nm flat gain) [8, 9]. For the transmission of high bit rate information, narrow region ranging 1530–1610 nm spectra will be utilized, which is based on EDFA systems. Bismuth borate glasses are the most suitable host candidates for achieving efficient luminescence in the rare earth ions, such as, erbium ion, since the luminescence efficiency is strongly dependent on the phonon energy of the host matrix. Incorporation of Er ions at Bismuth site, reduces the phonon energy,
thereby reduces non-radiative transitions, which intern increases the radiative life time of host glasses. It is found that, bismuth/Erbium co-doped glasses enrich the emission intensity in the longer wavelengths region, thus, broadening of the amplification window for fiber amplifiers is achieved [10].

Therefore, in the present work, different concentration of erbium doped zinc-lead-bismuth-borate glasses are prepared using conventional melt-quenching technique. Different physical properties are calculated and also, the structural and optical properties are investigated through different characterization techniques. The Judd-Olfet theory [11, 12] is used to investigate the local environment of the erbium ion in the host glass matrix. The JO intensity parameters are determined for all the prepared glasses and further they are used to evaluate the transition probabilities, branching ratio, stimulated cross section, effective line width and gain band width. Finally, the experimental results were compared with results of theoretical Judd-Olfet calculations for the possible applications in the field of photonics.

2. Experimental

Glass samples of 20ZnO-20PbO-(20-x)Bi₂O₃-xEr₂O₃-40B₂O₃ (x = 0,0.1,0.5,1,2,3 mol%), labelled as BE0, BE01,BE05,BE1,BE2 and BE3 respectively, are prepared by melt quenching method. AR graded with 99.9%
purity of ZnO, Bi₂O₃, PbO, H₃BO₃ and Er₂O₃ are used as starting materials. The stoichiometric amounts are homogeneously mixed, transferred to Porcelain crucible and then kept in furnace for 1 h at 1130 °C to obtain homogeneous melt. The melt then poured on to pre-heated brass mould and then suddenly quenched in the air atmosphere to obtain pellets. The prepared glasses are further annealed for 2 h at 350 °C, to remove thermal stress and strain present in the samples.

The density of glasses is estimated by Archimedes’s principle using toluene as an immersion liquid. Refractive index is determined using Abbe refractometer, mono-bromo-naphthalene as contact liquid. X-ray diffraction measurements are carried out using RIGAKU, ULTIMA IV x–ray diffractometer using Cu Kα radiation of wavelength 1.5418 Å to investigate the amorphous nature of the prepared glass samples. DTA studies are carried out in the temperature range 100 °C–800 °C by Perkin Elmer, USAA, Diamond TG-DTA instrument using Alumina crucibles of having 45 μl volume in the N₂ atmosphere with heating rate of 10 °C min⁻¹ having sensitivity of 0.06 μV. FTIR spectroscopic studies are carried out by KBr pellet method using Thermo Nicolet 6700 instrument in the range 500 to 1800 cm⁻¹. Optical studies are carried using UV–vis-NIR Double beam Spectrophotometer using Perkin–Elmer Lambda 750 instrument in the range 200–2000 nm for absorption with a UV–vis resolution of 0.17 to 5 nm and NIR 0.2 to 20 nm. Further, luminescence and time decay measurements are carried out using EDINBURGH FLS 980, using an excitation of 980 nm laser diode to record IR range with sensitivity >25 000:1 standard with life time range of 100 ps to 50 μs and with a pulse width <1 ns.
3. Results and discussion

3.1. Physical parameters
The density of the prepared glasses is determined by Archimedes principle using toluene as an immersion liquid. The refractive indices are estimated by digital Abbe refractometer instrument. Using the experimentally obtained density and refractive index values different physical properties such as, molar volume, molar refractivity, molar polarizability, polaron radius, inter ionic distance, field strength, dielectric constant, reflection loss and erbium ionic concentrations are estimated using suitable expressions available in the literature [13–17] and are summarized in table 1. It is observed that, the density and refractive index exhibits non-linear variation with erbium concentration. The other physical properties of the prepared glasses vary with increase in erbium ions concentration, indicates the distorted environment around the doped RE ion in the glass matrix. Figure 1 depicts the variation of different physical parameters with concentration of erbium in the host glasses. Incorporation of erbium ions in to the host glass matrix, result in losing the bonds which in turn results in the creation of non-bridging oxygen’s. The field strength found increasing with erbium concentration, while inter ionic distance decreases. Substitution of erbium ions causes change in oxygen to boron ratio, which induces BO\(_4\) units, results in the closely packed structure of the glasses, as evidences in the decrease in the molar volume values. The observed variations in the physical parameter results confirm the possibility of increase in the glass transition temperature in the prepared glass samples.

3.2. X-ray diffraction studies
The XRD patterns of the prepared glass samples are depicted in figure 2. It is observed from figure 2, that, there are no distinct crystalline peaks except two broad humps in the 2\(\theta\) region 20\(^{\circ}\)–40\(^{\circ}\) and 40\(^{\circ}\)–60\(^{\circ}\) within the detection limit of x-ray diffraction. The observed two humps are due to the short range order of atoms of the material and are the characteristics of amorphous nature of the glasses, which confirms the prepared glasses are in amorphous in nature.

Figure 4. (a) FTIR-Transmission and (b) FTIR-Absorption spectra of BE series glasses.
Table 2. FTIR spectrum bands assignment in wave number (cm$^{-1}$) for glass system.

| Sample                  | BE0 | BE01 | BE05 | BE1 | BE2 | BE3 | ASSIGNMENTS                                                                 |
|-------------------------|-----|------|------|-----|-----|-----|----------------------------------------------------------------------------|
| IR stretching/bending vibration band positions | 703 | 692  | 698  | 696 | 696 | 697 | Combined vibrations of BO$_4$ and PbO$_4$ groups or BiO$_6$ groups          |
|                         | 892 | 894  | 893  | 894 | 897 | 899 | Stretching vibrations of tetrahedral BO$_4$ units                           |
|                         | 1002| 999  | 997  | 1000| 994 | 1000| Pentaborate group                                                           |
|                         | 1236| 1231 | 1233 | 1232| 1282| 1281| B–O bond stretching vibrations and B–O bridging between B$_2$O$_3$ and BO$_3$ triangles |
|                         | 1361| 1380 | 1358 | 1379| 1374| 1358| Presence of pyroborate, orthoborate groups containing BO$_3$                |
3.3. Thermal studies

The DTA plots of the prepared glasses are depicted in the figure 3. The DTA graphs exhibits an exothermic peak in the range 650 °C–670 °C, which represents onset of crystallization temperatures (T_x) and an endothermic peak in the range 430 °C–450 °C, which represents the glass transition (T_g) temperature respectively. From DTA graph, the T_g and T_x are determined by taking inflection of dips (endo-down) and humps (exo-up) of the graph respectively.

Having determined values of T_g and T_x, further, the thermal stability factor, \( \Delta T = T_x - T_g \) is determined by taking the difference of two values. For BE0 T_g, T_x and \( \Delta T \) found to be 439 °C, 657 °C and 218 °C. Similarly for BE1 and BE3 the values of T_g, T_x and \( \Delta T \) found to be 441 °C, 670 °C, 229 °C and 456 °C, 688 °C, 232 °C respectively. The estimated values of, T_g, T_x and \( \Delta T \) are found increases with erbium concentration, indicates the stability of the glasses. That is the stability of the prepared glasses increases with increase of Er concentration in the host glass matrix. It is reported that, the stability of glass plays a vital role in deciding the suitability of glasses for photonic applications. Greater the stability, higher is the re-heating capacity \[16\]. Therefore, \( \Delta T \) must be maxima to accomplish fiber drawing glass, which will result in low optical absorption and scattering losses over long optical path lengths. Since, the prepared glasses exhibit greater thermal stability with increase in the Er concentration could be potential candidates for optical fiber applications \[17,18\].

3.4. Fourier transform infra-red spectroscopy

The composition dependent structural changes and the structural groups are investigated using Fourier transform infrared spectroscopy. The figures 4(a) and (b) shows the transmittance and absorbance spectra of BE series glasses. It is observed from the figure 4 that, three major bands are observed and the corresponding peak positions are summarized in the table 2 \[7,19\]. The bands observed around 700 cm\(^{-1}\) are attributed to

![Figure 5. Absorbance spectrum of BE1 glass sample.](image-url)

| Table 3. Summary of the wave number for free aqua ion (\( \nu_a \)), wave number for glass (\( \nu_c \)), experimental (\( \ell_{exp} \)) and calculated (\( \ell_{cal} \)) oscillator strengths, average nephelauxetic ratio (\( \beta \)), bonding parameter (\( \delta \)), root mean square deviation value (\( \Delta_{rms} \times 10^{-6} \)) and Judd–Ofelt parameters (\( \Omega_\lambda \times 10^{-20} \text{ cm}^2 \)). |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Wavelength      | 8 Transitions   | \( \nu_a \)     | \( \nu_c \)     | \( \ell_{exp} \) | \( \ell_{cal} \) | \( \beta \)      | \( \delta \)     | \( \Delta_{rms} \) | \( \Omega_2 \)    | \( \Omega_4 \)    | \( \Omega_6 \)
| 1529            | \( ^4I_{15/2} \) | 6540            | 6600            | 3.45            | 3.385           | 0.9970          | 0.2927          | \( \pm 0.641 \)  | 12.122          | 3.583           | 3.086           |
| 974             | \( ^4I_{11/2} \) | 10 267          | 10 250          | 1.054           | 1.636           |                 |                |                 |                 |                 |                 |
| 797             | \( ^4I_{9/2} \)  | 12 547          | 12 400          | 0.677           | 0.833           |                 |                |                 |                 |                 |                 |
| 652             | \( ^4F_{9/2} \)  | 15 337          | 15 250          | 5.311           | 5.230           |                 |                |                 |                 |                 |                 |
| 543             | \( ^4S_{9/2} \)  | 18 416          | 18 350          | 1.283           | 1.281           |                 |                |                 |                 |                 |                 |
| 521             | \( ^2H_{11/2} \) | 19 194          | 19 150          | 20.4            | 20.362          |                 |                |                 |                 |                 |                 |
| 488             | \( ^4P_{7/2} \)  | 20 492          | 20 450          | 4.78            | 5.141           |                 |                |                 |                 |                 |                 |
| 450             | \( ^4F_{5/2} \)  | 22 222          | 22 100          | 3.23            | 1.561           |                 |                |                 |                 |                 |                 |

\( \Delta_{max} \)
\( \Omega \)
\( \beta \)
\( \delta \)
\( \Delta_{rms} \)
\( \Omega_2 \)
\( \Omega_4 \)
\( \Omega_6 \)
combined vibrations of BO$_4$ and PbO$_4$ groups or BiO$_6$ groups. The bands in the region 800 to 1100 cm$^{-1}$ are attributed to the stretching vibrations of tetrahedral BO$_4$ units and pentaborate groups. The bands in the region 1100 to 1550 cm$^{-1}$ are attributed to the B–O bond stretching vibrations and the presence of borate groups. The summary of the same is given in Table 2. Clear observation shows that, the full width half maximum and peak positions are unaffected with the incorporation of erbium concentration in the host matrix. While the intensity of the peak shows non-linear trend with erbium concentration. The observed structural modifications are attributed to the incorporation of relatively less ionic sized Erbium doped compared to bismuth in the host glass matrix, which results in the creation of non-bridging oxygen’s. Due to the creation of NBO’s the BO$_4$ units show increase in intensity.

3.5. Absorption studies
All the BE series glass samples shows similar absorption spectra and number of transitions as well. Further to evaluate the intensity and JO parameters, the absorption spectrum for BE1 sample in UV–vis–NIR region is depicted in Figure 5 and the all parameters are evaluated for all the samples. From Figure 5, eight absorption peaks corresponds to the transitions from the ground state $^4$I$_{15/2}$ of rare earth ion (Er$^{3+}$) are observed in case of BE1 glass. The observed transition (absorption peaks) are compared with standard aqueous Er$^{3+}$-ion [20]. Among all the observed transitions, the transition from $^4$I$_{13/2}$ to $^2$H$_{11/2}$ is having highest intensity, considered as the hypersensitive transition, which is characteristic of the host dependent. Using absorption spectrum, the intensity parameters are determined. Further, using Judd-Olfet theory [11, 12], the experimentally calculated line strength values are compared with the theoretically obtained values and the estimated values are summarised in Table 3. It is observed that, the JO parameters follows trend $\Omega_2 > \Omega_4 > \Omega_6$. From the Table 3, it is concluded that, $\Omega_2$ is greater, bonding parameter is +ve and the erbium ions have covalency bond nature with the host. Hence compared to previous reported [21–24] literature, our matrix is having high covalency nature.

Using these values, the radiative properties such as, radiative transition probability ($A_R$), branching ratio ($\beta_R$) and radiative lifetimes ($\tau_R$) are estimated for $^4$I$_{13/2} \rightarrow ^4$I$_{15/2}$ transition, the corresponding values are 341.83.
100% and 2.925 ms respectively. Furthermore, the F-L theory [25] is used to estimate the simulative cross section ($\sigma(\lambda_p)$) and effective bandwidth ($\Delta \lambda_{\text{eff}}$) of emission band theoretically and their obtained values are $\sigma(\lambda_p) = 8.489 \times 10^{-21}$ cm$^2$ and $\Delta \lambda_{\text{eff}} = 99$ nm respectively.

3.6. Luminescence and life time studies

The Luminescence studies of BE series glasses is carried out in IR range using excitation wavelength of 980 nm, corresponds to $^4I_{13/2} \rightarrow ^4I_{15/2}$ transition and the obtained spectra is depicted in the figure 6(a). From figure 6(a), broad emission spectrum is observed in all samples. It is observed that, the intensity of the emission peak increases with erbium ions concentration up to 1 mol% and there after decreases for further increase in erbium ions concentration. That is, the luminescence enhancement is seen with erbium ions concentration initially till 1 mol% and further increase in the Erbium ions concentration, spectra shows luminescence quenching effect. The observed quenching effect is due to concentration quenching effect [21–25]. The time decay studies are carried out for same $^4I_{13/2} \rightarrow ^4I_{15/2}$ transition and are shown in figure 6(b). From figure 6(b), all the samples, show single exponential behaviour, which makes easier and simpler to estimate the life time of each sample. All the samples life time is fitted and the obtained values are depicted in figure 6(b). The observed decay curves also confirms the concentration quenching effect as result the life time values found decreasing with increase in erbium concentration. Further, the amplification parameters such as, the gain bandwidth ($\Delta G = \Delta \lambda_{\text{eff}} \times \sigma(\lambda_p)$) and gain per unit length ($G = \tau_{\text{exp}} \times \sigma(\lambda_p)$) are estimated and the corresponding values are found to be $840 \times 10^{-28}$ cm$^3$ and $3.989 \times 10^{-24}$ cm$^3$s respectively, which are greater than the previous reported values [26].

3.7. McCumber’s theory and gain coefficient

Using McCumber’s theory [27], the absorption ($\sigma_{\text{abs}}(\lambda)$) and emission ($\sigma_{\text{emi}}(\lambda)$) cross section values are estimated and the corresponding plots are shown in figure 7(a). The estimated values from graph of both $\sigma_{\text{abs}}(\lambda)$

![Figure 7. (a) The absorption and emission cross-section spectra and (b) Gain co-efficient for $^4I_{13/2} \rightarrow ^4I_{15/2}$ transition of Er$^{3+}$ ion doped BE1 glass.](image-url)
Table 4. For $^1_{13/2} \rightarrow ^1_{15/2}$ emission transition 1 mol% Er$^{3+}$ ions doped glasses.

| Matrix | $\lambda_p$ (nm) | FWHM (nm) | $A_p$ (s$^{-1}$) | $\sigma$ (×10$^{-21}$ cm$^2$) | $\Delta G$ (×10$^{-28}$ cm$^3$) | $\tau_r$ (ms) | $\beta$ (%) | Optical gain (×10$^{-24}$ cm$^2$ s) | $\sigma_e$ (10$^{-21}$ cm$^2$) | $\sigma_a$ (10$^{-21}$ cm$^2$) |
|--------|-----------------|-----------|-----------------|-----------------|-----------------|--------------|----------|-------------------------------|----------------------------|-----------------|
| BE1 (Present work) | 1531 | 99 | 341.83 | 8.489 | 840 | 2.925 | 100 | 3.39 | 5.666 | 6.378 |
| 50 B$_2$O$_3$-20 TeO$_2$-10SrO-10CaF$_2$-9 Bi$_2$O$_3$ [28] | 1572 | 80 | 441.05 | 14.72 | 117.8 | 2.26 | 100 | 33.4 | 4.92 | 4.89 |
| 44P$_2$O$_5$ + 17K$_2$O + 29 ZnF$_2$ + 9Al$_2$O$_3$ [29] | 1536 | 31 | 177.18 | 17.79 | 160.9 | 5.64 | 100 | — | 5.19 | 4.32 |
| 64P$_2$O$_5$ + 13Bi$_2$O$_3$ + 10Li$_2$O + 10Na$_2$O [26] | 1534 | 38 | — | 8.74 | 332 | 5.78 | 100 | 15.29 | 8.54 | 8.98 |
| 30 Li$_2$O-20 LiF-44 B$_2$O$_3$-5 ZnO [30] | 1532 | — | 168 | — | — | 2.53 | 100 | — | — | — |
| 44P$_2$O$_5$-17 K$_2$O-9Al$_2$O$_3$-23 PbO-6Na$_2$O [31] | 1534 | 46 | 144 | 6.73 | 310 | 6.91 | 100 | 13.86 | 6.69 | 5.9 |
| 39B$_2$O$_3$-10SiO$_2$-10Al$_2$O$_3$-30ZnO-10Li$_2$O [32] | 1536 | 53.9 | 925 | 2.63 | 563 | 1.09 | 100 | — | — | — |
and $\sigma_{\text{rms}}(\lambda)$ respectively are $5.666 \times 10^{-21}$ cm$^2$ and $6.378 \times 10^{-21}$ cm$^2$. Further, for different population inversion, the absorption and emission cross sections are plotted as a function of wavelength to estimate the approximate value of population inversion and the corresponding spectra is shown in figure 7(b). From figure 7(b), it is observed that, there is a 40% population inversion in case of BE1 glass and it originates in the telecommunication C band region [21–27].

3.8. Comparison of optical properties

Optical properties of 1 mol% erbium doped glass is considered and compared with the different hosts [26, 28–32]. The overall values and deciding factors for application purpose are summarized in the table 4. For $^4I_{15/2} \rightarrow ^4I_{13/2}$ emission transition, the peak wavelength, FWHM, radiative transition probability, stimulated emission cross-section, gain band width, radiative life time, branching ratio, optical gain, absorption and emission cross section values by FL theory is considered. Among the all host, our sample has FWHM 99 nm, which is suitable for optical fiber applications.

4. Conclusions

In the present investigation, Erbium doped zinc-lead-bismuth-borate glasses are prepared using the conventional melt quenching technique. Results confirm that, the obtained glasses show high density and refractive index due to the presence of heavy metal oxides in the matrix. The prepared glasses are found amorphous in nature and shows high thermal stability. The FTIR spectra exhibits major borate related (BO$_4$ and BO$_3$ units) vibration modes and their increase in intensities are observed due to the creation of NBO’s after incorporation of erbium in the matrix. The absorption spectra of BE series in the UV–vis-NIR region shows 8 transition states. Among the observed peaks, the peak corresponds to the $^1I_{15/2} \rightarrow ^3H_{11/2}$ is found highest intensity called hyper sensitive transition (HST). Further, the covalency nature of the BE glasses is revealed from J0 intensity and bonding parameters. The radiative probabilities shows highest branching ratio for $^4I_{15/2} \rightarrow ^4I_{11/2}$ transition. The luminescence spectra confirms the enhancement of luminescence at excitation of 980 nm up to 1 mol% Er concentration in the host matrix and further increase of Er concentration beyond 1 mol%, luminescence quenching effect is observed, which is due to the cross relaxation channels present in the glass matrix. The emission cross-section values are estimated using FL and MC theory and the estimated values found in agreement with absorption cross-section values from the spectral broadening, as well as from the overlap nature of absorption and emission spectra. In addition, 40% of population inversion (C band) is observed in BE glass. The investigated result confirms the Er doped zinc-lead-bismuth-borate glasses are suitable candidates for long-range telecommunication broad band optical amplifier applications.

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