Magnesium Fluoride Borate Glasses for Low Phonon Energy

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Abstract
An oxyfluoroborate glass series of composition 75B2O3-5Al2O3–(20-x) Li2O–xMgF2 (where x = 0, 5, 10, and 15 mol.%) was prepared using the normal melt-quenching technique. The physical properties (density, molar volume, and different optical behaviors) of the glass system were investigated via different techniques and discussed with the substitution of Li2O for MgF2. Also, the absorption coefficient, both direct and indirect optical energy gaps, and the optical exciton energy gap were studied. Furthermore, IR spectroscopy was used as a structural probe of the nearest-neighbor environment in the glass network. The results elucidate that the replacement of Li2O with MgF2 leads to both a blueshift in absorption cutoff and a decrease in the direct energy gap. More liberation of Mg2+ ions produces more localized states during transition, which decreases the values of band gap energy. The addition of MgF2 has a clear impact on lowering the glass phonon energy, which makes this glass promising for fiber amplifiers that operate at certain telecommunications wavelength bands and for upconversion fiber lasers. These results show the capability of using oxyfluoroborate glass series to be applicable in optical amplifier laser components.

Keywords Magnesium borate glass · phonon energy · absorption coefficient

Introduction
Recently, the development of photonic materials has become more significant for the progress of photonics applications and the telecommunications industry.1–3 Upconversion is the mechanism that converts low-energy photons into visible light.4 The most effective upconverters must consist of host systems that provide low non-radiative losses so that radiative emission (luminescence) could be enhanced. Different glass hosts such as borate, phosphate, germanate, vanadate, and tellurite families have been extensively studied for this objective.5 Among these families, borate glasses are preferred in certain optical, optoelectronic and photonic devices mainly owing to their (i) low working temperatures, (ii) wide glass forming range, (iii) second- and third-order optical non-linearity6 and borate glasses are most consistent for high concentrations of transition metal and rare earth ion doping.7 The borate glasses modified by various constituents such as alkali oxide, alkaline-earth oxide, aluminum oxide, fluorides, and heavy metal oxides have the advantages of high transparency, high thermal stability, low phonon energy compared to that of pure borate glass, which is high (1400 cm−1).8,9 The oxyfluoride borate glasses are one of the highly efficient upconversion luminescence hosts; they not only have higher chemical and mechanical stability but also lower phonon energy.10 The modifier oxides influence the high covalent strength B-O bonds, creating the so-called borate anomaly (the existence of different anionic tri-coordinate borate and four-coordinate borate species). The increase of the disorder degree of the glass structure results in a high density of localized states in the allowed energy band tails. This gives the glass its optoelectronic properties.11 Since the photonic property is related to the ionic character of the glass network, it is necessary to tune the structure of glass to enhance the conversion of covalent to ionic conditions to adjust the energy band gap of glass. Accordingly, the effect of MgF2 on lithium alumino-borate glass is studied for upconversion applications.

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In the field of developing the glass structure system by embedding rare earth elements or transition metals for enhancing their phonon and optical characteristics to be used in optoelectronic applications, In 2013, Zhou et al. studied Er$^{3+}$-doped fluorotellurite glasses to be applied in the development field of optical laser amplifier components as this structure leads to emission of infrared radiation (1.32 μm) due to the phonon characteristic of the fluorotellurite glass structure. In 2018, Qi et al. succeeded in preparing a new series of fluoroaluminophosphate glasses based on the structure of fluoroaluminate with varying amounts of Cl−, which showed a novel phonon characteristic. This perspective was discussed as the increase of chlorine ions in this kind of glass structure leads to an increase in the thermal stability of their host structure, which allows them to be applied in aser materials. In 2019, Marzouk et al. studied the effect of doping MnO$_2$ and MoO$_3$ on two different glass matrixes from sodium phosphate glasses, which affected the UV and absorption characteristics, followed by developing of its optical energy band gap values due to formation of modifier groups from manganese ions, molybdenum ions, and phosphate ions. In 2020, Al-Mokhtar et al. found that the addition of MgF$_2$ to some borate glass increases non-bridging oxygen, followed by increasing the disorder of its system, which affected the development of its optical properties by generation of a transmission band at the short wavelength side and an absorption edge at the long wavelength side; consequently, as the embedding of MgF$_2$ in the glass matrix increases, its energy ΔE increases. In 2021, El-Daly et al. synthesized and investigated the optical characteristics of cobalt borate glass, which showed an increase in phonon characteristics as the amount of embedded Co increased due to the interatomic spacing effect in the prepared glass structure.

In this work, an oxy-fluoro borate glass series of composition 75B$_2$O$_3$-5Al$_2$O$_3$-(20-x) Li$_2$O-xMgF$_2$ (where x = 0, 5, 10, and 15 mol.%) was prepared using the normal melt-quenching technique to study the change in their optical characteristics. The effect of MgF$_2$ on the optical features of the main glass structure was investigated by transition and vibration absorption spectroscopy. This study was carried out to test the capability for using the prepared glass system material in the development field for optical laser amplifier or optical fiber.

### Methodology

#### Preparation of Glass Samples

The chemical composition of 75B$_2$O$_3$-5Al$_2$O$_3$-(20-x) Li$_2$O-xMgF$_2$ (where x = 0, 5, 10, and 15 mol.%) is designed to prepare a series of glass samples by melt-quenching. The starting materials are reagent grade chemicals of H$_3$BO$_3$, Al$_2$O$_3$, Li$_2$CO$_3$ and MgF$_2$. Certain amounts of these chemicals are mixed well and melted at 1000°C in a porcelain crucible for 1 h in an electric glow-bar furnace. Continuous stirring was done during the melting to ensure complete homogeneity. The melts are then poured rapidly into a stainless-steel mould of defined dimensions and annealed in a preheated furnace at a temperature of 400°C and left to cool gradually to room temperature at a rate of 30°C/h to relieve any internal mechanical stresses. The shaped samples are polished well to measure their physical and optical properties.

### Characterisation

The FTIR spectra of the studied glasses were recorded by a computerized FTIR spectrometer (type Bruker VERTEX 8V, Germany) covering the wavenumber range of 400–4000 cm$^{-1}$ with a resolution of 4 cm$^{-1}$. Spectroscopic measurements: A UV/VIS/NIR spectrophotometer (Jasco V-770, Japan) was used to collect the optical absorption and reflectance spectra for the samples at ambient conditions. The density of glass samples ($\rho$) is measured by the Archimedes rule, and xylene is the immersion liquid, using the next formula,

$$\rho = \frac{W_A}{W_A - W_X} \rho_x$$

where $W_A$ is the weight of samples in air while $W_X$ is the weight of samples in liquid. The molar volume $V_m$ of glasses was calculated by using Eq. (2):

$$V_m = \sum x_i \frac{M_i}{\rho}$$

where $x_i$ is the molar fraction and $M_i$ is the molecular weight of the ith component of the studied compositions.

### Results and Discussion

#### Density and Molar Volume Calculation

Firstly, supplementary Figure S1 depicts the X-ray diffraction patterns of a series of MgF2-containing glasses. The findings show that all of the synthesized glasses are amorphous, with no hint of crystalline phases. The density (glass) and molar volume ($V_m$) values of the 75B$_2$O$_3$-5Al$_2$O$_3$-(20-x)Li$_2$O-xMgF$_2$ glass samples with increasing substitution amounts of MgF$_2$ are displayed in Fig. 1. The measured values of density show an anomalous behavior, as depicted in this figure. As boosting the glass network with 5 mol.% of MgF$_2$ at the expense of Li$_2$O increased the density value, a decrease to a minimum
density value versus the increase of the MgF₂ ratio to 10 mol.% is observed. This anomalous behavior is followed by a new increase in density value as the molar fraction, x, rises to its maximum value at 15 mol.% However, enhancing the glass network with substituting amounts of MgF₂ gradually increased the molar volume values. Molar volume is more sensitive to structural differences between glasses than density because it normalizes for atomic weights of different glass components. The observed increase in VM demonstrates that the packing of the coordination polyhedral in the structural glass matrix of the alumino-fluoroborate glasses improves with increasing MgF₂ content.

The anomalous behavior of density can be explained as follows. The structure of the present glass network depends mainly on the two construction units of the borate network, BO₃⁰ (normal triangle coordination) and BO₄⁻ (fourfold tetrahedral coordination) that are linked together via oxygen atoms to create B-O-B bonds. Firstly, the modifying action of MgF₂ converts part of BO₃⁰ into BO₄⁻. This conversion process increases the amount of bridging oxygen (BO) and consequently increases the connectivity and compactness of the studied glass network. Moreover, the addition of MgF₂ breaks up the local symmetry of the structural BO₄⁻ units and converts them into neutral structural units BO₃⁰. Therefore, MgF₂ can form –O–Mg–O and Mg–O–B bonds, creating an open structure with a greater number of BO₃⁰ units than BO₄⁻. This conversion creates coordinated non-bridging oxygen (NBO) (oxygen dangling bond) defects. The maximum addition of MgF₂ modifier to the studied borate network provides the network with excess Mg ions. The big difference between the atomic weights and densities of the modifier Mg²⁺ is (At. W = 24.305 u, ρ = 1.738 g/cm³) and Li⁺ is (At. W = 6.941 u, ρ = 0.534 g/cm³). Hence, Mg²⁺ ions are more efficient at filling the interstitial spaces in the glass network structure than Li⁺ ions, leading to the formation of a more compact network.

FTIR Spectroscopic Measurements

Figure 2 illustrates the structural FTIR vibrational bands of the studied glasses. It is clear that there are four band regions. The first is in the far infrared range of about 400–500 cm⁻¹, and the second is cited at about 700 cm⁻¹. The two other bands are broad and extend from 800–1200 cm⁻¹ and 1200–1400 cm⁻¹, respectively. By increasing MgF₂ content, unremarkable shifting for the bands is cited in the near IR region, while the band at about 700 cm⁻¹ remains as it is. The strong changes are remarkable at the two broad band regions 800–1200 cm⁻¹ and 1200–1400 cm⁻¹, where more broadness is detected at 800–1200 cm⁻¹ and increasing intensities at bands observed at 1200–1400 cm⁻¹. More data can be observed in Fig. 3, where the deconvoluted graphs split the broad band into several sharp bands. These sharp bands are listed in Table I.

From all three IR figures, it can be recognized that the bands cited at 424 and 472 cm⁻¹ are related to the vibration of Li⁺ ions, while the band at 461 cm⁻¹ which formed after the addition of MgF₂ is due to the vibrations of Mg²⁺ cations in their sites. A bending vibrational absorption band
centered at about 690 cm\(^{-1}\), which is correlated to B–O–B.\(^{19}\) Three bands observed at about 806, 920 and 1063 cm\(^{-1}\) are related to the stretching vibrations of BO\(_4\) tetrahedral borate groups.\(^{20}\) After replacing Li\(_2\)O by MgF\(_2\), these three bands were shifted to about 901, 1011, and 1099 cm\(^{-1}\), respectively. These band changes can be interpreted as the formation of MgO\(_4\) groups instead of BO\(_4\).\(^{21}\) Finally, the bands at about 1227, 1360 and 1473 cm\(^{-1}\), which are related to the symmetric and asymmetric stretching vibrations of boroxol groups BO\(_3\), are shifted to 1227, 1435 and 1506 cm\(^{-1}\), and the formation of a new absorption band at about 1344 cm\(^{-1}\) is caused by the increase of Mg\(^{2+}\) ions in the glass system.\(^{21}\)

**Sub-Band Gap Structural Properties**

The study of the transmittance and reflectance of the samples with different composite structures as a function of different wavelengths is shown in Fig. 4a,b. From Fig. 4a, the glass transmittance of the main structure of 75B\(_2\)O\(_3\)·(20-x)Li\(_2\)O·5Al\(_2\)O\(_3\)·xMgF\(_2\) was increased from 53% to 77%, 65%, and 67% for the addition of 5, 10, and 15 mol.% of MgF\(_2\), respectively. This significant change in the transmittance value is related to the incorporation of MgF\(_2\) in different amounts. The amount of addition of MgF\(_2\) was affected to increase the transmittance optical properties of the main glass structure of 75B\(_2\)O\(_3\)·20Li\(_2\)O·5Al\(_2\)O\(_3\) to 24%, 12%, and 14% when the molar amounts were changed to be 5, 10, and 15 mol.%. According to the traditional polarization theory related to the transmittance spectral line, the increase in the transmission spectral line may be related to the addition of the MgF\(_2\) structure instead of Li\(_2\)O with its characteristic broad energy band gap and high bonding strength. Also, each spectral line is responsible for an individual characteristic absorption cutoff wavelength as the main glass structure with any molar percentage addition of MgF\(_2\) has a 371-nm absorption cutoff wavelength. This value showed a blueshift to a higher wavelength after the addition of different molar

![Deconvoluted IR spectra of (a) base glass (b) MgF-3.](image-url)

**Table 1** The peak positions and their vibrational modes of the high MgF\(_2\) glass structure

| Peak position | Vibrational mode                           | Peak position | Vibrational mode                           |
|---------------|--------------------------------------------|---------------|--------------------------------------------|
| Base glass    |                                            | High MgF\(_2\) glass |                                            |
| (cm\(^{-1}\)) | (cm\(^{-1}\))                              | (cm\(^{-1}\)) | (cm\(^{-1}\))                              |
| 424           | Li\(^{+}\)-O                                | 461           | Mg-O-Mg                                    |
| 472           |                                            | 598           |                                            |
| 563           | Bending vibrations of various borate units  | 692           | Oxygen bridge between two trigonal boron atoms |
| 690           | Oxygen bridge between two trigonal boron atoms | 901           | Diborate groups                            |
| 802           | Tri-, tetra- and pentaborate groups         | 1011          |                                            |
| 920           | Diborate groups                             | 1099          | Tri-, tetra- and pentaborate groups         |
| 1063          | Tri-, tetra- and pentaborate groups         | 1227          | Pyro- and orthoborate units                 |
| 1223          | Pyro- and orthoborate units                 | 1344          | B–O vibrations of various borate rings      |
| 1360          | B–O vibrations of various borate rings      | 1435          | B–O\(^{+}\) bonds                          |
| 1470          | B–O\(^{-}\) bonds                          | 1506          |                                            |
percentages of MgF$_2$ were 426, 410, and 415 for the addition of 5, 10, and 15 mol.% of MgF$_2$, respectively. This change is related to the change in the molecular weight of the main glass structure under the effect of embedding of MgF$_2$ instead of the number of molecular structures from Li$_2$O.

From the transmittance (T) and reflectance (R) of the different prepared structural forms of the main glass structure, the absorbance coefficient of the glass ($\alpha$) can be calculated from the Beer-Lambert law:

$$\alpha = \frac{1}{L} \ln \left( \frac{1 - R^2}{2T} + \sqrt{\frac{R^2 + (1 - R^2)^2}{4T^2}} \right)$$

where $L$ is the thickness of the glass sample. The absorbance and transmittance investigations depend on changes in the energy band gap values and type of transition. The value of the energy band gap and the type of transition (direct or indirect) can be detected based on the Tauc relation.

$$\alpha h\nu = B(h\nu - E_g)^n,$$

where $\alpha$ is the absorption coefficient, $h\nu$ is the discrete photon energy, $B$ is a constant, $n$ is the type of transition (2 in the case of direct transition or $\frac{1}{2}$ in the case of indirect transition), $E_g = h\nu$ is the optical energy band gap, and $\omega$ is the frequency. By plotting the relation between $(\alpha h\nu)^2$ versus $E_g$, the detection of the energy band gap from the direct transition was detected. It showed that the energy band gap of the direct transition for the main glass structure after its embedding is 4.13 eV, while this value was changed to be 3.67, 3.91, and 3.87 eV for the addition of 5, 10, and 15 mol.% of MgF$_2$, respectively, as shown in Fig. 4c. In other words, the relationship between $(\alpha h\nu)^{0.5}$ versus $h\nu$ represents the detection of energy band from an indirect transition. It is clear that the energy band gap for the main glass structure after its embedding is 1.64 eV, while this value was changed to be 2.69, 2.31, and 2.58 eV for the addition of 5, 10, and 15 mol.% of MgF$_2$, respectively, as shown in Fig. 4d. From Fig. 4c–d, it is clear that there is a variation between the energy band gap in the case of direct transition in compared with indirect transition. In addition, the variation of the energy band gap has the same type of transition when the molar ratio of the main glass structure was changed, which is related to the embedding process of the main glass structure by decreasing the molar amount of Li$_2$O and increasing the molar amount of MgF$_2$ leading to the enhancement of the polymerization process, which allowed the appearance of more localized states during transition affected by the values of the energy band gap.$^{22,23}$ So it was concluded that...

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Fig. 4 (a) UV-Visible transmittance spectra, (b) UV-Visible reflectance spectra, and the energy band gap investigation from (c) indirect transition and (d) direct transition of the glass structure of $75B_2O_3.(20-x)Li_2O.5Al_2O_3.xMgF_2$. 

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the energy band gap increased with increased embedding of MgF₂ in the matrix glass, which is related to the transition produced from the HOMO levels and LUMO levels in their crystal structure. When the fluoroborate glass structure was transferred to alkali borate and alkali fluoroborate glass structure, BO₄ units with excess amounts were generated. The LUMO level of these alkali structures consisted of vacant orbitals caused by oxygen atoms of borate oxide structure and the energy band gap of pure borate oxide structure is around 8 eV. When the MgF₂ structure started to be embedded, the HOMO level started to be reduced, and consequently, caused a decrease of the energy band gap value (as mentioned for 5 mol.% of MgF₂), and was followed by increasing the energy band gap value as the amount of fluorine increased (as mentioned for 10 mol.% of MgF₂) due to the effect embedding of fluorine. After that, the energy band gap started to decrease again as the amount of fluorine further increased (as mentioned for 15 mol.% of MgF₂) due to the creation of non-bridging Mg, which bound excited electrons less firmly as the bridging Mg increases with MgF₂, leading to a reduction in the energy band gap value.¹⁵,²⁴

Furthermore, the previously calculated energy band gap appearing in Fig. 4c–d could be represented by the exciton energy band gap ($E_{\text{gap-ext}}$), which could be used to inform the optical energy band gap ($E_{\text{gap-opt}}$) of the glass structure before and after the addition of MgF₂ by studying the 1st derivative of the optical absorbance values of the different glass structures as a function of photon energy values as shown in Fig. 5a. The maximum detection peak in each spectrum could be represented by the optical energy band gap ($E_{\text{gap-opt}}$). The changes of the optical energy band gap compared with the exciton energy band gap could be confirmed from Fig. 5b.²⁵,²⁶

According to the network structure of the glass formation, the optical transition was changed, which affected the energy band gap transition either directly or indirectly. The behavior of the changes in the optical frequencies molecular of the main glass structure was changed as the amount of MgF₂ was embedded, which is represented by phonon energy. The phonon energy ($E_\Omega$) could be calculated based on the following formula:

$$E_\Omega = \frac{E_{\text{opt}}^d - E_{\text{opt}}^\text{ind}}{2}$$

where $E_{\text{opt}}^d$ and $E_{\text{opt}}^\text{ind}$ are the energy band gap from direct transition and indirect transition of the glass structure of 75B₂O₃.(20-x)Li₂O.5Al₂O₃.xMgF₂ before and after the addition of MgF₂ with different molar percentages of MgF₂, respectively. The factor 2 in the phonon energy calculation is related to the presence of two electrons in each momentum-energy. From this calculation, the phonon energy of the glass structure of 75B₂O₃.(20-x)Li₂O.5Al₂O₃.xMgF₂ before and after the addition of MgF₂ with different molar percentages of MgF₂ as a function of the molar ratio of MgF₂/(LiO₂ + MgF₂) was studied as shown in Fig. 6. From this figure, it is clear that the glass structure has low phonon energy, which allows it to be applied in fiber amplifiers.¹¹,²⁷,²⁸

**Conclusion**

A novel oxyfluoroborate glass is prepared with the replacement of Li₂O by MgF₂ intentionally to reduce the glass phonon energy. An increase in glass molar volume ($V_M$) demonstrates that the packing of the coordination polyhedra in the structural glass matrix of the studied glasses improves with increasing MgF₂ with liberation of Mg⁺² ions that are more able to fill the interstitial spaces in the glass network structure than Li⁺ ions leading to the formation of more compact network. FTIR deconvoluted spectra confirmed the role of Mg⁺² on the structural network with successive increases of MgF₂ until 15 mol.%.

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![Fig. 5](image-url) (a) The first derivative of the absorbance spectra, and (b) the changes in the optical band gap and the exciton energy of the different glass structures with different molar percentages of MgF₂.

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optical energy band gaps are differently affected with the implanted MgF₂, while the glass phonon energy is decreased from 1.2 eV to 0.6 eV, which makes the designed new system promising for fiber amplifiers that operate at certain telecommunications wavelength bands and for upconversion fiber lasers.

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