



Strontium ions concentration dependent modifications on structural and optical features of $\text{Li}_4\text{Sr}(\text{BO}_3)_3$ glass



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ABSTRACT

Composition optimization is essential for achieving improved properties of strontium (Sr^{2+}) doped lithium borate (LSBO) glass. Glasses with compositions $(85-x)\text{H}_3\text{BO}_3 + 15\text{Li}_2\text{CO}_3 + x\text{SrCO}_3$, where $x = 0.5 - 2.5$ mol% are synthesized via melt quenching technique. Glasses are characterized to determine Sr^{2+} ions concentration dependent improvements in the structural, physical, and optical properties. XRD patterns verifies the amorphous nature, FESEM images reveals transmitting surface morphology, stable glasses with Hurby parameter ~ 0.5 are achieved. FTIR spectra revealed three fundamental peaks around $700.55-930.68$ and 1072.07 cm^{-1} , which corresponded to the trigonal and tetrahedral stretching vibrations of BO_3 and BO_4 units. These peaks are shifted with the increase of modifier concentration. Energy for Urbach tail, direct and indirect optical gaps are calculated. The increasing glass refractive index from 2.34 to 3.00 is ascribed to the conversion of BO_4 into BO_3 units. Emission spectra exhibit three peaks centered at 492.65, 536 and 549 nm, corresponding to the transitions from the $^1\text{D}_2$, $^3\text{D}_3$ and $^3\text{D}_2$ levels to ground state $^1\text{S}_0$ respectively, and accompanied by a slight blueshift attributed to the formation of new complexes. Effects of strontium ion contents on the structural and optical properties are demonstrated. Our observation may contribute towards the development of LSBO based glass photonics.

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1. Introduction

Synthetic and natural borates have potential for several industrial applications. Their excellent products including boric acid, anhydrous boric, anhydrous borax, borax pentahydrate, borax decorate, borax decahydrate, and sodium perborate in recrystallized units are highly demanding. To obtain a better borate products and comprehending the structural chemistry of borate crystals remain the key issue [1]. Despite being bit hygroscopic, borate glass possesses various striking properties such as effective atomic number, high transparency, low melting point, and high-temperature stability [2,3]. Naturally lithium borates do exist as white powder of melting point of 917 °C, solubility between 1 and 10% and density of 2.4 g cm^{-3} . Borate crystals are quite prospective for their notable piezoelectric properties [4]. In the past, several alkaline and alkali earth metals are incorporated as a modifier to change the borate glass structures [5]. Their excellent chemical durability, thermal stability, and the most stable active ion hosts

make borate systems attractive in optics and as sensors [6,7]. The increased release of electrons together with the lowering of the hygroscopic properties of borate glass [8–11] due to the incorporation of strontium ion is reported. Despite much research, the Sr^{2+} assisted improvements in the structural, and optical properties are not clearly understood.

According to the best of the authors' knowledge, for the first time the effect of Sr^{2+} ion concentration variation on the lithium-borate glass network structure and subsequent modification of their optical properties are systematically scrutinized. We present the improvements in structural and optical characteristics of LSBO glass under varying concentration of the SrCO_3 modifier. The influence of Sr^{2+} ion concentrations on the structure and the optical response is systematically analyzed via sample synthesis and thorough characterizations. A relationship between structural changes, modification in the optical responses and Sr^{2+} concentration variation is demonstrated.

2. Materials and methods

Series of $\text{Li}_4\text{Sr}(\text{BO}_3)_3$ glasses having chemical compositions $(85-x)\text{H}_3\text{BO}_3 + 15\% \text{Li}_2\text{CO}_3 + x\text{SrCO}_3$ ($x = 0.5, 1, 1.5, 2$ and 2.5 mol%)

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were synthesized via conventional melt-quenching method. A milling machine was used to get a fine powder of the compounds after it was weighted and well-mixed. The mixture was melted in an alumina crucible for 1 h using an electric furnace a NabGmbH at emperature of 1300 °C. The Li_2CO_3 (purity 99.+)%, H_3BO_3 (purity 99.98%) and SrCO_3 (purity 99.9%) were supplied by Syarikat Pustaka Elit, Johor Bahru, Malaysia. Upon melting, the liquid mixture was proud and quenched on well-polished pre-heated steel mould. Annealing was carried out at 400 °C for 3 h to minimize any residual stress. Five samples (A1, A2, A3, A4 and A5) with their compositions are enlisted in Table 1.

The amorphous nature of all samples was verified using X-ray diffraction (XRD) (Siemens Diffractometer D5000), with Cu K_α radiation ($\lambda = 1.54 \text{ \AA}$). The particle morphology, purity and the phase homogeneity of these glasses were analyzed via field emission scanning electron microscopy (FESEM). The details of elemental traces were detected from EDX measurements. Infrared spectra of prepared samples, the wave number range of $4000\text{--}400 \text{ cm}^{-1}$ were recorded using a Scimitar FTS 2000 instrument (FTIR). Potassium bromide (KBr) pletteingmethod with 100:1 mg ratio was used for FTIR measurements at instrument resolution of 0.8 cm^{-1} . The mixture was then pressed at 120 MPa pressure to achieve clear sample of dimension $\sim 2.0 \text{ mm} \times 10.0 \text{ mm}$. Differential thermal analysis (DTA) was performed on Perkin Elmer Pyris Diamond Analyzer equipped with a copper target and nickel filter. The heating rate of $10 \text{ }^\circ\text{C}/\text{min}$ over temperature region $50\text{--}1000 \text{ }^\circ\text{C}$ was applied.

The room temperature absorption measurement of synthesized samples in the wavelength range of $200\text{--}2000 \text{ nm}$ was performed via a UV–Vis–NIR spectrophotometer (Shimadzu 3101). The absorbance signal was analyzed using diffraction grating with a double monochromator and R-928 photomultiplier detector (resolution $\sim 0.1 \text{ nm}$). Physical parameters including density (ρ), molar volume (V_m), refractive index (n), molar refractivity (R_m), polaron radius (r_p), inter-nuclear distance (r_i), field strength (F), ion concentration (N), and Urbach energy were also determined. Emission measurement was performed via Perkin Elmer LS55 Luminescence Spectrophotometer interfaced with a xenon arc lamp of wavelength $300\text{--}1300 \text{ nm}$ as excitation source. The luminescence signal was recorded via Monk-Gillieson monochromator and a photodiode was to record the signal.

3. Results and discussion

Fig. 1 displays the XRD patterns of synthesized LSBO samples at various concentrations of SrO. The complete absence of any sharp peaks confirms the amorphous nature [12,13]. Additionally, the appearance of two peaks around $20\text{--}30^\circ$ and $40\text{--}50^\circ$ are due to LiBO_3 and SrBO_3 respectively, attributed to the amorphous structure of glasses.

Fig. 2 illustrates the FESEM images of all samples. Clear homogeneous morphology in the absence of any grain is manifested. Fig. 2 shows every FESEM image that rightly detect the elements'

Table 1
Sample compositions and code.

Samples code	Concentration (mol %)		
	Li_2CO_3	SrCO_3	H_3BO_3
A1	15%	0.5%	84.5%
A2	15%	1%	84%
A3	15%	1.5%	83.5%
A4	15%	2%	83%
A5	15%	2.5%	82.5%

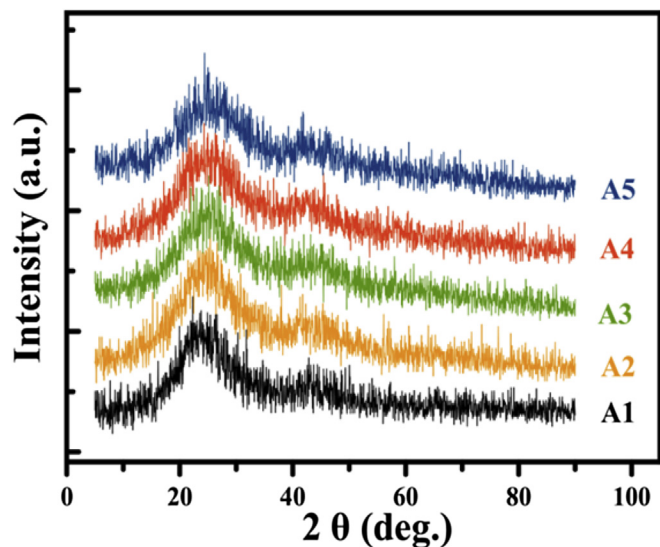


Fig. 1. SrO concentrations dependent XRD patterns of LSBO glasses.

morphology. The structure of glass is found to alter with the increase of SrO concentration. These structural changes are verified through the FTIR measurement.

Fig. 3 displays the IR spectra of all prepared glasses, where three major absorption bands are evidenced [14–16]. Three fundamental peaks observed in the range $700.55\text{--}930.68$, 1072.07 cm^{-1} are assigned to trigonal and tetrahedral stretching vibrations of the network. Furthermore, the peak shifted toward a lower frequency with the increase of modifier Sr^{2+} concentration are attributed to the high atomic weight of strontium than other atoms in the network [17]. The modes at 700.55 , 696.16 , 696.16 , 696.16 and 687.39 cm^{-1} are assigned to B–O–B stretching vibration of trigonal structure of BO_3 and BO_4 groups [18,19], intensity of this band decreases with an addition of SrCO_3 . The bond observed within $890.19\text{--}930.68 \text{ cm}^{-1}$ and $1054.18\text{--}1072.07 \text{ cm}^{-1}$ are assigned to stretching vibration of B–O bond of BO_4 groups [20–22]. A compact structure with a higher density is expected because BO_4 groups are tightly bonded as compared to the BO_3 groups [23]. The appearance of strong bond around $1200\text{--}1500 \text{ cm}^{-1}$ originated from B–O symmetric stretching of BO_3 groups of ortho-borate units [24,25]. Overall, the intensity of all bands are modified due to the addition of SrCO_3 contents. The occurrence of broad bands in the region of $3400\text{--}4000 \text{ cm}^{-1}$ are assigned to the O–H groups (H–O–H) symmetric stretches [26,27]. FTIR band assignments are listed in Table 2. The source of error associated to the measurement are mostly instrumental and within the experimental range $\sim 5\%$.

The temperature for glass transition, crystallization, melting, and sublimation are determined from DTA analyses. Material under study together with inert reference material is subjected to the same thermal condition, and changes between two materials are recorded. Glass forming ability is estimated via Kauzmann relation [13],

$$T_{rg} = \frac{T_g}{T_m} \quad (1)$$

where T_g is the glass transition temperature and T_m is the glass melting temperature. Thus, glass forming ability is said to be good if $0.5 \leq T_{rg} \leq 0.66$ is satisfied. For the present glass composition Kauzmann assumption is obeyed with excellent values of T_{rg} . The glass thermal stability is determined using the following Hurby

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