Synthesis and Optical Investigations on (Ba, Sr)TiO$_3$ Borosilicate Glasses Doped with La$_2$O$_3$

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ABSTRACT

In this research paper we are reporting synthesis, structural and optical investigations of barium strontium titanate borosilicate glasses with addition of La$_2$O$_3$. Glasses were synthesized by conventional rapid melt quench method. Infrared absorption spectra, for various (Ba, Sr)TiO$_3$ borosilicate glass samples having glass system 64[(Ba$_{1-x}$Sr$_x$)TiO$_3$]-35[2SiO$_2$-B$_2$O$_3$]-5[K$_2$O]-1[La$_2$O$_3$] (x = 0.3, 0.5, 0.6, 0.8 and 1.0), were recorded over a continuous spectral range from 450 - 4000 cm$^{-1}$. IR spectra were analyzed to determine and differentiate the various vibrational modes in the structural changes. Raman spectroscopy of all glass samples were also carried out wavenumber range form 200 - 1500 cm$^{-1}$. These two complementary spectroscopic techniques revealed that the network structure of the studied glasses is mainly based on BO$_3$, pentaborate groups linked to BO$_4$ tetrahedra and units placed in different structural groups, the BO$_3$ units are dominating. The recorded IR and Raman spectra of different glasses are used to clarify the optical properties of the prepared glass samples correlating with their structure and compositions. UV-Vis spectroscopy was carried out in range of 200 - 800 nm. The optical band gap was found between 2.023 - 3.320 eV.

Keywords: (Ba, Sr)TiO$_3$; Infrared Spectroscopy; UV-Vis Spectroscopy; Raman Spectroscopy

1. Introduction

Glasses are defined as inorganic product of fusion which has been cooled to a rigid condition without crystallization without crystallization [1]. The main distinction between glass and crystals is the presence of long range order in the crystal structure. A widespread set of very different borate glasses with optical, magnetic, superionic conductivity and other technologically interesting properties are currently produced. The optimization of such properties as a function of compositions and other synthesis parameters are required a good knowledge of the microscopic glassy structure. For many years, glasses containing transition metal ions have attracted attention because of their potential applications in electrochemical, electronic and electro-optic devices [2]. IR spectroscopy is one of the most common spectroscopic techniques used by organic and inorganic chemists. Simply, it is the absorption measurement of different IR frequencies by a sample positioned in the path of an IR beam. The main goal of IR spectroscopic analysis is to determine the chemical functional groups of the samples. Using various sampling accessories, IR spectrometers can accept a wide range of types of sample such as gases, liquids, and solids. Thus, IR spectroscopy is an important and popular tool for structural elucidation and compound identification [3]. Raman spectroscopy is also an essential tool for characterization of the structure, environment, and dynamics of glassy materials. Furthermore, the portability of the technique allows for its use in on-line process monitoring [4]. In Raman spectroscopy, the nature of the light matter interaction is not same as in IR spectroscopy and the fundamental differences between the two processes determine the selection rules, which control Raman or IR activity of normal mode of vibrations. Interaction of IR radiation with a normal mode of vibration only occurs when the electric field of radiation oscillates with same frequency as instant dipoles caused by atomic vibrations. A normal vibration can be IR active only if a change in the dipole moment of the vibration occurs and is a one photon process, as only photon is absorbed [5]. Therefore, IR spectra give addition information than Raman spectra by which the symmetries of normal...
modes of vibration of molecules and crystal lattices are determined [6-8]. Addition of various alkali modifiers, \( A_2O \) (A: Li, Na, K, Rb, Cs), to the borate glasses brings determined [6-8]. Addition of various alkali modifiers, structural units are described by reaction given below:

\[ 2(BO_3^-) + 2e^+ \rightarrow 2\left(BO_2^+\right) + 2A^+ \]  
\[ 2(BO_3^-) + 2e^+ \rightarrow 2\left(BO_2^+\right) + 2A^+ \]

The existence of four coordinated boron in alkali-doped borate glasses has been studied extensively for a wide variety of modifiers through various spectroscopic techniques such as IR, Raman, NMR, and NQR spectroscopy [9,10]. Recently, our group reported few publications on structural, crystallization and dielectric studies of \((\text{Pb, Sr})\text{TiO}_3\) borosilicate glass and glass ceramics. These studies shows very high dielectric constant and refractive index and their glass ceramics shows the sluggishness of the crystallization in glass matrix due to doping of \(\text{La}_2\text{O}_3\) [14,15].

2. Experimental Procedure

2.1. Sample Preparation

High purity analytical reagent grade chemicals \(\text{BaCO}_3\) (Himedia 99%), \(\text{SrCO}_3\) (Himedia 99%), \(\text{TiO}_2\) (Himedia 99%), \(\text{SiO}_2\) (Himedia 99.5%), \(\text{H}_2\text{BO}_3\) (Himedia 99.8%), \(\text{K}_2\text{CO}_3\) (Himedia 99.9%), \(\text{La}_2\text{O}_3\) and (Himedia 99.9%) were used for the preparation of various glass samples having glass system \(64[(\text{Ba}_1-x\text{Sr}_x)\text{TiO}_3]-30[2\text{SiO}_2-\text{B}_2\text{O}_3]-5[\text{K}_2\text{O}]-[\text{La}_2\text{O}_3](x = 0.3, 0.5, 0.6, 0.8 and 1.0)\). Appropriate amounts of raw materials, as per the composition of glasses, were properly weighed and mixed in an agate mortar using acetone as mixing medium and dried the powder. The glass batches of 20 grams were melted in a programmable electric furnace. The melting temperatures for different compositions were in the range 1050°C - 1450°C. The melt was maintained at the melting temperature in the furnace for 10 minutes for refining and homogenization. The melt was poured into an aluminum mould and pressed by a thick aluminum plate then immediately transferred in to a preheated muffle furnace for annealing at temperature 450°C for 3 hours to remove the residual stresses due to temperature gradient, which is produced by rapid cooling. The glasses were cooled to room temperature within the furnace after annealing.

Nomenclature of prepared glass samples contains six letters (BSTKL and T) and four digit groups (5, 1 and 0.3 to 1.0 digit numbers). First three letters BST refers to barium strontium titanate borosilicate. The term 5K designates the 5% of \(\text{K}_2\text{O}\) while term L indicates, whether \(\text{La}_2\text{O}_3\) is used as donor dopants and the numeric term before L denotes molecular percentage of \(\text{La}_2\text{O}_3\). The last numeric number denotes the content of strontium or composition, x value.

2.2. Infrared Spectroscopy

The powdered glass samples were mixed with KBr powder and pressed as pellets. Then, these pellets are used as samples for recording the IR spectra. The IR spectra of BST borosilicate glasses are carried out using JASCO FT/IR-5300 in the wave number range 450 - 4000 cm\(^{-1}\) at room temperature.

2.3. Raman Spectroscopy

The powdered glass samples are used as in the wave number range from 200 - 1500 cm\(^{-1}\). Micro Raman setup, Renishaw, UK, equipped with a grating of 1800 lines/mm and Olympus (model MX-50) A/T was attached with spectrometer which focuses laser light into sample and collect the scattered at 1800 by scattering geometry. The 15.4 nm \(\text{Ar}^+\) laser was used as an excitation source and GRAM-32 software for data collection.

2.4. UV-Vis Spectroscopy

UV-visible absorption spectroscopy is a very useful technique to characterizing the optical and electronic properties of different materials such as thin films, filters, pigments and glasses. Measurement of the optical band gap of glass sample is carried out using the data within range 200 - 800 nm obtained by spectrophotometer. UV-vis spectroscopy of the samples was carried out using UV-visible spectrophotometer (Varian, Carry-50Bio). It measures the percentage of radiation in the different regions such as ultra-violet (200 - 400 nm) and visible (400 - 800 nm) regions that is absorbed at each wavelength within ultra-violet and visible regions. Optical transitions are basically two types, direct and indirect transitions. In these transitions, the electromagnetic radiations interact with the electrons in the valence band and which reaches to conduction band by gaining fundamental band gap. These transitions occur in both crystalline and amorphous semiconductor materials. These transitions are related with Mott and Davis relation. For photon energies just above fundamental edge, the relation between ab-

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The absorption coefficient ($\alpha$) and photon energy is given below [16,17]:

$$\alpha = \left( \frac{\hbar v - E_g}{\hbar v} \right)^n$$  \hspace{1cm} (3)

where $A$ is a constant related to the extent of the band tailing, $n = 1/2$ for allowed direct transition, $n = 2$ for allowed indirect transition, $\hbar v$ is the photon energy and $E_g$ is the optical band gap energy of the material. The absorption coefficient, $\alpha$, was calculated at different photon energies by using the relation, $\alpha = A/d$, where $A$ is the absorbance and $d$ the thickness of the samples. The optical band gap, $E_g$, was calculated by extrapolating the linear parts of the curves to $\left( \frac{\alpha \hbar v}{\alpha} \right)^n = 0$ of curve $\left( \frac{\alpha \hbar v}{\alpha} \right)^n$ versus $\hbar v$ and extrapolating for the glass samples.

2.5. Density Studies of Glass Samples

The density strongly depends on the composition and structure of the samples. Density of the glass samples were determined by liquid displacement method of Archimedes principle [18]. Distilled water was used as the liquid medium. Density of glass and glass ceramic samples were calculated using the formula:

$$\rho = \frac{(W_2 - W_1)}{(W_4 - W_3 - W_1)}$$ \hspace{1cm} (4)

$\rho$ = Density (gram/cc);
$W_1$ = Weight of empty specific gravity bottle (gram);
$W_2$ = Weight of specific gravity bottle with sample (gram);
$W_3$ = Weight of specific gravity bottle with sample and distill water (gram);
$W_4$ = Weight of specific gravity bottle with distill water (gram).

3. Results and Discussion

3.1. Infrared Spectroscopy

The IR spectra for various glass samples of BST borosilicate glasses doped with La$_2$O$_3$ are shown in Figure 1. IR spectra of all glass samples consist of various absorption bands in different regions lies between wavenumbers 450 - 4000 cm$^{-1}$. These bands are influenced by doping the variation in content of Ba/Sr ratio because the positions of some absorption bands are shifted due to compositional variations. Wavenumbers of different absorption peaks for all the glass samples have been listed in Table 1. The first absorption band lies in wavenumber range 3440 - 3470 cm$^{-1}$. The position of this broad band slightly shifted towards lower wavenumber side with increasing the concentration of Sr. This absorption band occurs due to molecular water inside the glassy network [19]. Absorption bands were observed in the wavenumber range 2853 - 2924 cm$^{-1}$ and these absorption bands are attributed to formation of hydrogen bonding [20-22]. These bands are almost unaffected by the variations of Ba/Sr ratio. An IR spectrum of these glass samples also shows diffused absorption bands in wavenumbers range 2340 - 2365 cm$^{-1}$. The doublet splitting was observed in this band and attributed to -OH bonding vibrations which are formed at non-bridging oxygen sites and hydroxyl groups are usually present in borate glasses. The presence of -OH groups may due to the KBr pellet technique used to record IR spectra [23]. There are few absorption bands in the wavenumber range 1275 - 1739 cm$^{-1}$ are observed due to the asymmetric stretching relaxation of the B-O bonds of trigonal BO$_3$ units. Such types of vibrational modes were observed within wavenumbers range from 1200 - 1750 cm$^{-1}$ [24,25]. The band at 1560 cm$^{-1}$ was absent in all glass samples except glass samples BST5K1L0.5 and BST5K1L0.6. The bands near wave-number 1737 cm$^{-1}$ are present only in barium rich glass.
Table 1. Peak positions in IR spectra of glass samples in the system 64\[(Ba_{1-x}Sr_x)TiO_3\]-30\[2SiO_2.B_2O_3\]-5 \[K_2O\]-1\[La_2O_3\].

| Glass samples code | Wavenumber of different absorption peaks (cm$^{-1}$) |
|-------------------|------------------------------------------|
|                   | 1          | 2          | 3        | 4          | 5 | 6 | 7 | 8 | 9 |
| BST5K1L0.3        | 3470       | 2924       | 2853     | 2365       | 2342 | - | 1343 | 983 | 707 | 467 |
| BST5K1L0.5        | 3480       | 2923       | 2853     | 2365       | 2340 | 1560 | 1332 | 984 | 707 | 467 |
| BST5K1L0.6        | 3450       | 2923       | 2853     | 2365       | 2348 | 1560 | 1340 | 992 | 719 | 479 |
| BST5K1L0.8        | 3450       | 2923       | 2853     | 2365       | 2342 | -    | 1332 | 991 | 719 | 467 |
| ST5K1L1.0         | 3440       | 2924       | 2854     | 2364       | 2346 | -    | 1356 | 992 | 707 | 464 |

Table 2. Peak positions in Raman spectra of different glass samples in the glass system 64\[(Ba_{1-x}Sr_x) TiO_3\]-30\[2SiO_2.B_2O_3\]-5 \[K_2O\]-1\[La_2O_3\].

| Glass sample code | Raman band positions (cm$^{-1}$) |
|-------------------|----------------------------------|
|                   | 1          | 2          | 3        | 4 |
| BST5K1L0.3        | 977       | 812       | 675     | 248 |
| BST5K1L0.5        | 979       | 829       | 708     | 285 |
| BST5K1L0.6        | 980       | 836       | 698     | 275 |
| BST5K1L0.8        | 979       | 833       | 720     | 281 |
| ST5K1L1.0         | 981       | 803       | 670     | 248 |

Figure 2. Raman spectra of BST borosilicate glass samples: (a) BST5K1L0.3, (b) BST5K1L0.5, (c) BST5K1L0.6, (d) BST5K1L0.8 and ST5K1L1.0.

3.2. Raman Spectroscopy

Raman spectra of different BST borosilicate glass samples doped with La$_2$O$_3$ are shown in Figures 2(a)-(e) and the peak positions of Raman bands have been listed in Table 2. Each Raman spectra indicate four different kinds of peaks. Raman pattern of glass sample BST5K1L0.3 is shown in Figure 2(a). It shows four broad and overlapped peaks at different wavenumbers 248, 675, 812 and 977 cm$^{-1}$. The Raman band at wavenumber 977 cm$^{-1}$ was occurred due to B-O$^-$ stretching in orthoborate units [30]. The peak at 675 cm$^{-1}$ is the characteristic of B-O-B stretching in metaborate rings [31,32] while peak at wavenumber 812 cm$^{-1}$ occurs due to symmetric breathing vibrations of boroxol rings [33,34]. Raman bands of glass
sample BST5K1L0.5 shows four bands at various wave-numbers 285, 708, 829 and 979 cm$^{-1}$ respectively (Figure 2(b)). The Raman band near 829 cm$^{-1}$ is attributed to B-O-B stretching in pyroborate units [31,32]. Figure 2(c) depicts the Raman spectra of glass sample BST5K1L0.6 and showing three distinct peaks with different intensities at wavenumbers 275, 698, 836 and 980 cm$^{-1}$. These Raman bands were also present in glass samples BST5K1L0.8 and BST5K1L1.0 with slight shifting in their wavenumbers (Figures 2(d)-(e)). The non linear variations were observed with changing the Ba/Sr ratio in glassy matrix. When the content of Sr was increased from 0.6 to 0.8 or 1.0, the band at 836 shifted towards lower wavenumber side. This may be due to non uniform variation in symmetry and dipole moment during the measurements. The weak Raman peak were observed their spectra due to metallic cations towards low wavenumber side. The assignment of IR and Raman bands in the spectra of different glass samples are summarized in Table 3.

3.3. UV-Vis NIR Spectroscopy

UV-visible absorption spectra of various (BaSr)TiO$_3$ borosilicate glass samples have shown in Figure 3. The band gap, $E_g$ for BST borosilicate glasses have been listed in Table 4.

There is not any sharp increase in absorption at energies closed to the band gap that manifests itself as an absorption edge in the UV-visible absorption spectra and it is indicating the amorphous nature of glass samples. It is observed that absorption edges shifted towards higher wavelength side with to increasing concentration of SrO. For pure Sr glass sample of composition, a drastic increase in its absorption edge at increasing the content of SrO and which shows red shift due 580 nm, which shows the translucency inside glass sample. The indirect optical band gap of BST borosilicate glasses are determined by using Davis and Mott relation as discussed in section 2.4. The plot of $h\nu$ versus $(\alpha h\nu)^{1/2}$ shown in Figure 4. The optical band gap was found to be in range of 2.023 - 3.320 eV. The similar results were also reported on Ba-TiO$_3$ by Suzuki [35]. The band gap decreases with increasing the concentration of SrO. This shows the composition dependence of optical band gap [36,37]. The pure Sr content glass sample has lowest band gap value 2.023 eV. This sudden difference for band gap value may be due to the variation of non-bridging oxygens. The shift of the absorption edge to a higher wavelength or decrease of $E_g$ with increasing SrO content may be due to more porous nature of Sr rich glass samples than that of Ba rich glass samples, and also increased the oxygen amount inside the samples. Hence, an increase in the formation of bridging oxygen (BO$_4$ units) and which makes the sample more semiconducting [38].

3.4. Density Studies

The density of BST borosilicate glasses was found between 2.55 - 2.84 gm/cc. It decreases with increasing concentration of SrO in the BST borosilicate glasses while it increases with increasing the concentration of La$_2$O$_3$. This may be due to high density of Ba (3.51 g/cc) and Sr (2.61 g/cc). Almost linear trend was observed in the density of glasses following equation $y = 3 - 0.46x$ and this was shown in Figure 5. The values of density of BST borosilicate glasses have been listed in Table 4.

4. Conclusion

Perovskite bulk transparent BST borosilicate glasses were prepared successfully by rapid melt quench method. The IR and Raman bands were found to be composition dependent. IR spectra of BST borosilicate glass samples shows that borate is major network former in this glass system and it is also confirmed by Raman spectroscopic studies. The vibrations due to metallic cations (Ba$^{2+}$ and Sr$^{2+}$) are also play important role as a network modifier at lower wavenumber sides in their IR as well as Raman spectra. The optical band gap shows slight dependence on compositions and it was found lowest for the glass sample ST5K1L1.0 (2.023 eV).

Table 3. Assignment of infrared and Raman bands in the spectra of different glass samples.

| Wave number (cm$^{-1}$) | IR assignments | Raman assignments |
|-------------------------|----------------|------------------|
| 471 - 523               | 248 - 285      | Vibrations of metal cations such as Ba$^{2+}$, Sr$^{2+}$ |
| 722 - 828               | 670 - 720      | bonding of B-O-B linkages (diborate linkage) |
| 1035                    | 803 - 812      | stretching vibration of B-O-Si linkage |
| 1200 - 1635             | 820 - 836      | asymmetric stretching relaxation of the B-O bond of trigonal BO$_3$ units |
| 2292 - 2376             | 977 - 981      | -OH bonding |
| 2700 - 3000             | -              | Hydrogen bonding |
| 3391 - 3435             | -              | Molecular water |

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Figure 3. UV-visible absorption spectra of all BST boro-silicate glass samples.

Figure 4. Davis and Mott plot of \( \alpha \) versus \( (\alpha h \nu)^{1/2} \) of glass sample (a) BST5K1L0.3, (b) BST5K1L0.5, (c) BST5K1L0.6, (d) BST5K1L0.8 and (e) ST5K1L1.0.

Figure 5. Variation of density vs content of Sr.

Table 4. Optical band gap and density of glass samples in the system 64[(Ba\(_{1-x}\)Sr\(_x\)).TiO\(_3\)]-35[2SiO\(_2\).B\(_2\)O\(_3\)]-5[K\(_2\)O]-1[La\(_2\)O\(_3\)].

| Compositions (x) | Glass codes | Optical band gap (eV) | Density (gram/cc) |
|------------------|-------------|----------------------|------------------|
| 0.3              | BST5K1L0.3  | 3.320                | 2.84             |
| 0.5              | BST5K1L0.5  | 3.315                | 2.80             |
| 0.6              | BST5K1L0.6  | 3.311                | 2.775            |
| 0.8              | BST5K1L0.8  | 3.307                | 2.6              |
| 1.0              | ST5K1L1.0   | 2.023                | 2.55             |

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