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Determination of Optical Band Gaps and Structural Properties of Cu²⁺ Doped B₂O₃-Na₂O-Al₂O₃-V₂O₅ Glasses

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ABSTRACT

Two different groups of compositions for $80B_2O_3 + 15Na_2O + (5-x) Al_2O_3 + (x) V_2O_5$ and $85B_2O_3 + 10Na_2O + (5-x) Al_2O_3 + (x) V_2O_5$ (where x=1; 0.5 wt %) glass samples were prepared by quenching technique and doped with Cu^{2^+} ions. Optical and structural properties of undoped glasses, and glasses doped with Cu^{2^+} ions were examined. Their structural properties were determined with SEM images and EDX spectra. Optical band gaps of glass samples were determined for direct and indirect transitions from the transmittance spectra. Through the fundamental ultra violet absorption edges of the glasses, the optical band gap energies and Urbach energies were calculated to be within the range of 2.963–3.228 eV for direct and 2.592–2.807 eV for indirect transitions. The physical properties of all glasses were also evaluated with respect to the composition.

Key Words: Glasses, Vanadium, Semiconductors, Optical Properties.

1. INTRODUCTION

Glasses have many technological applications due to their electrical and optical properties. Vanadium doped glasses are known to demonstrate semiconductor properties.

Borate glasses are the focus of interest due to their structures and properties that are quite different from silica glasses. Many researchers have studied B_2O_3 intensively in recent years [1].

Borate glasses are used as electro-optic switches, electro-optic modulators, solid-state laser materials and non-linear optical parametric converters [2-4]. In addition, they are often used as dielectric and insulating materials and it is known that borate glass constitutes a good shield against IR radiation [5]. Studies on glasses containing relatively low concentration of V_2O_5 in presence of B_2O_3 are very few [6].

Semiconducting transition metal oxide (such as V_2O_5)based glasses have gained much interest in solid-state chemistry and materials science with regard to their possible applications as memory and switching devices [7-11].

Vanadium-containing oxide glasses are known to be semiconductors and the transport mechanism involves the exchange of electrons between vanadium (IV) and vanadium (V) centers, e.g. [12]: $V^{4+} - O - V^{5+} \iff V^{5+} - O - V^{4+}$

Vanadate glasses are identified as n-type semiconductors for low V^{4+}/V^{5+} ratio [11]. It is also known that V^{5+} in low ratios enter the amorphous structure as an impurity whereas V^{5+} in high ratios are present in the structure as glass formers [13].

Some authors studied the effect of single [14, 15] and multiple [16] TM (transition metal) ions as dopant in alkali and alkaline earth oxide glasses [17]. Glasses doped with TM ions came into prominence because of their notable spectroscopic properties and their suitability for fiber optic communications, luminescent solar energy concentrators (LSCs) [18].

In this study, we studied the absorption and transmission of B_2O_3 . $Na_2O \cdot Al_2O_3 \cdot V_2O_5$ (CuO) glasses prepared in two groups. Optical band gaps and Urbach energies for direct and indirect transitions of each group were calculated, defects of the surfaces and their structural properties were determined by SEM and EDX spectra.

2. EXPERIMENTAL

2.1. Sample Preparation

Glass samples belonging to two groups were prepared with Alfa Aeser brand chemicals and their compositions were given in Table 1. Reagents were > 99.9% pure.

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Quenching technique was used for the preparation of glass samples.

Table 1. Compositions of the glasses (all in wt %) studied in the present work.

| Batch I | |
|------------|--|
| Glass Code | Glass Structure |
| BNAVI-1 | $80B_2O_3{\cdot}15Na_2O{\cdot}4Al_2O_3{\cdot}1V_2O_5$ |
| BNAVI-2 | 80B ₂ O ₃ ·15 Na ₂ O ·4,5Al ₂ O3·0.5V ₂ O ₅ |
| BNAVCuI-1 | $80B_2O_3 \cdot 15Na_2O \cdot 4Al_2O_3 \cdot 1V_2O_5 : 0.5CuO$ |
| BNAVCuI-2 | 80B ₂ O ₃ ·15Na ₂ O·4.5Al ₂ O ₃ ·0,5V ₂ O ₅ :0.5CuO |
| Batch II | |
| Glass Code | Glass Structure |
| BNAVII-1 | $85B_2O_3 \cdot 10Na_2O \cdot 4Al_2O_3 \cdot 1V_2O_5$ |
| BNAVII-2 | $85B_2O_3 \cdot 10Na_2O \cdot 4.5Al_2O_3 \cdot 0.5V_2O_5$ |
| BNAVCuII-1 | 85B ₂ O ₃ ·10Na ₂ O·4Al ₂ O ₃ ·1V ₂ O ₅ :0.5CuO |
| BNAVCuII-2 | $85B_2O_3{\cdot}10Na_2O{\cdot}4.5Al_2O_3{\cdot}0.5V_2O_5{\cdot}0.5CuO$ |

Glass samples of the first group had the composition of $80 \text{ B}_2\text{O}_3 + 15 \text{ Na}_2\text{O} + (5-x) \text{ Al}_2\text{O}_3 + x \text{ V}_2\text{O}_5$ and glass samples had the composition of 85 $B_2O_3 + 10 Na_2O +$ (5-x) Al_2O_3 + x V_2O_5 , (x=1; 0.5). At first, reagents were precisely weighed, then powdered and mixed. Mixtures were then transferred into a platinum crucible and kept in an electric furnace for 1 hour at 1673 K; molten glass samples were taken out with certain intervals and stirred to ensure homogeneity. After an hour, molten glass samples were taken and poured into a graphite mould to yield cylindrical glass blocks with 35 mm heights and diameters of 25 mm. In order to prevent breaks and cracks, these glass blocks were taken out of the mould, kept in the sintering furnace at 673 K for 30 minutes, and then cooled slowly to the room temperature. These glass blocks were cut by a cutting device, having a diamond disc, into 2.5 mm thick discs to facilitate measurement; these discs were ground and polished to give parallel and shiny surfaces. The glass samples were powdered in a porcelain mortar for XRD measurements.

2.2. Measurements

One of the most definite methods to determine whether a glass sample has an amorphous structure or not, involves the XRD measurements. X-ray diffraction patterns of the powdered samples were obtained by using a Shimadzu XRD–6000 model x-ray diffractometer using Cu x-ray tube between $2\theta=0^{\circ}-70^{\circ}$. Surface micrographs and distribution spectra were examined in order to determine the homogeneity and the defects of the structure. SEM-EDX measurements of all samples were conducted by JEOL/JSM-6335/INCA-EDS (Scanning Electron Microscope and Semi Quantitative Elemental Analysis System).

Measurements and images from two different regions of the sample were obtained for each sample and a healthier comment was made on the homogeneity of the samples.

Transmission and absorption spectra of each sample were measured with a HITACHI 150-20 Spectrophotometer using deuterium lamp at room temperature.

Optical band gaps and Urbach energies were calculated for direct and indirect transitions of each sample from UV-Visible absorption spectra.

3. RESULTS AND DISCUSSION

3.1. X-ray Diffraction

X-ray diffraction detects readily crystals in a glassy matrix if the dimensions of the crystals are greater than 100 nm [19].

X-ray diffraction of each glass system was examined separately and with the help of x-ray diffraction patterns, and information related to the structures of these glasses were obtained. Figure 1 presents the x-ray diffraction patterns of 4 undoped glasses.



Figure 1. X-ray diffraction patterns belonging to BNAVI-1, BNAVI-2, BNAVII-1, BNAVII-2 glass samples.

As it can be seen in Figure 1, diffraction patterns of all glass samples do not demonstrate any detectable peak. All these patterns indicate that glass samples have pure amorphous, non-crystalline structures. Two humps at $2\theta = 25^{\circ}$ and $2\theta = 45^{\circ}$ were also observed in previous studies of borate glass samples [19].

3.2. SEM Images and EDX Spectra

As it can be seen in Figures 2 (A) and (B), white spots are visible. These spots are considered to be the result of the interaction of glasses with atmospheric humidity, which is a typical behavior of borate glasses. These spots were not seen in SEM images given in Figures 3 (A) and (B). When the results of their EDX spectra were examined (Table 2), it was seen that percentage weight ratios of O and V elements obtained from the first region of BNAVI-1 glass were approximately equal to the ones obtained from the second region. Percentages of Na and Al elements varied in a very small proportion, and only in two regions. When the results of EDX spectra of BNAVII-1 were examined, it was seen that their percentage weight ratios were slightly different from each other. Apart from the measurements that have been presented here for two samples, similar results have been obtained from these processes for the other six samples, and it was understood with the measurements obtained from two different regions of each glass that an almost homogenous distribution was formed in the glass. With the testimony of the spots on the surface, it was concluded that, undoped BNAVI-1, BNAVI-2 glasses containing 80% B2O3 interact with air more than BNAVII-1, BNAVII-2 glasses containing 85% B2O3. In addition, it was observed that these defects increase in all Cu²⁺ doped glasses.



Figure 2. SEM image and EDX spectrum obtained from (A) the first region and (B) the second region of BNAVI-1 glass.



Figure 3. SEM image and EDX spectrum obtained from (A) the first region and (B) the second region of BNAVII-1 glass.

Table 2. EDX results of BNAVI-1 and BNAVII-1 glasses.

| | BNAVI-1 First Region | | BNAVI-1 Second Region | | BNAVII-1 First Region | | BNAVII-1 Second Region | |
|---------|----------------------|---------|-----------------------|---------|-----------------------|---------|------------------------|---------|
| Element | Weight% | Atomic% | Weight% | Atomic% | Weight% | Atomic% | Weight% | Atomic% |
| O-K | 77.28 | 84.09 | 77.07 | 84.00 | 89.62 | 93.30 | 88.85 | 92.91 |
| Na-K | 15.27 | 11.56 | 14.93 | 11.33 | 5.87 | 4.26 | 5.74 | 4.18 |
| Al-K | 5.94 | 3.83 | 6.38 | 4.12 | 3.34 | 2.06 | 3.89 | 2.41 |
| V-K | 1.52 | 0.52 | 1.62 | 0.55 | 1.17 | 0.38 | 1.51 | 0.50 |
| Totals | 100.00 | | 100.00 | | 100.00 | | 100.00 | |



3.3. Transmission and Absorbance Spectra

Figure 4. (a) Transmittance, (b) absorbance spectra of all samples versus wavelength.

Examination of transmittance spectra is important to study the color characteristics of the glass samples and the effects of transition elements in these formed glass systems. Vanadium ions are present in the formed glass structures and vanadium may give blue, turquoise, green, brown color depending upon the melting conditions and soda content in sodium borate glasses or give no color at all. Maxima, similar to soda-lime-silica glasses containing 0.5% V₂O₅ in the literature were not

seen in BNAVI-1, BNAVI-2, BNAVII-1, BNAVII-2 glasses [20-22].

Copper in sodium borate glasses [23] give a color ranging from green to turquoise in low soda values and a color ranging to turquoise in high soda content. Transmittance spectra of the glasses obtained in this study showed that, transmittance maxima varied from 484 and 497 nm and formed characteristic curves due to

cupric ions (Figure 4(a)). BNAVCuI-1, BNAVCuI-2, BNAVCuII-1 and BNAVCuII-2 have transmittance maxima of 489 nm, 497 nm, 484 nm and 489 nm, respectively.

UV absorption edges of absorbance spectra (Figure 4(b)) used for the determination of optical band gaps were determined to be 386 nm, 382 nm, 400 nm, 382 nm, 389 nm, 367 nm, 386 nm, 364 nm for BNAVI-1, BNAVI-2, BNAVII-1, BNAVII-2, BNAVCuI-1, BNAVCuI-2, BNAVCuII-1 and BNAVCuII-2 respectively.

3.4. Optical Band Gap and Urbach Energy

Analysis of optical spectra is one of the most beneficial tools to figure out the electronic structures of amorphous semiconductors [24]. Measurement of the optical absorption coefficient (α) near the fundamental absorption edge is particularly a standard method for the investigation of optically induced electronic transitions in many materials. Two types of optical transitions, i.e. direct and indirect, occur at the absorption edge [25,26]. The absorption as a function of wavelength for all compositions of glass is shown in Figure 4(b). The absorption coefficient, below and near the edge of each curve was determined at different wavelengths using relation (3.1);

$$\alpha(\nu) = \left(\frac{1}{d}\right) \ln\left(\frac{I_0}{I_t}\right)$$
(3.1)

where I₀ and I_t are intensities of the incident and transmitted beams, respectively and d corresponds to thickness of each sample. The factor $\ln(I_0/I_t)$ is the absorbance [6]. "d" thicknesses belonging to glass samples are given in Table 3. Optical band gaps were calculated using absorption spectra for direct and indirect transitions for all prepared glass samples.

For direct transitions (3.2);

1

$$\alpha(\nu) = B(h\nu - E_{opt})^n / h\nu$$
(3.2)

where n=1/2 for allowed transition, B is a constant and E_{opt} is direct optical band gap. Relation (3.2) is also used for indirect transitions. In this equation, where n=2for allowed transition, B is a constant and E_{opt} is indirect optical band gap. Using equations 3.2 and 3.3 and by plotting $(\alpha h v)^{1/2}$ and $(\alpha h v)^2$ as a function of photon energy hv, optical band gaps for indirect and direct transitions could be found, respectively. The respective values of Eopt were obtained by extrapolating $(\alpha h v)^{1/2} = 0$ for indirect transitions to and $(\alpha h v)^2 = 0$ for direct transitions [27].

Logarithm of the absorption coefficient was plotted as a function of photon energy for various compositions of batches I and II, respectively. Urbach energy values (ΔE) were calculated by taking the reciprocals of the slopes of linear portion in the lower photon energy region of these curves as stated with relation (3.3) [28].

$$\alpha(\nu) = \alpha_0 \exp(h\nu/\Delta E)$$
(3.3)

Direct and indirect optical band gap values and Urbach energies of all samples are given in Table 3.



Figure 5. $(\alpha h v)^2 \sim h v$ variations of glass samples in (a) Batch I, (b) Batch II.



Figure 6. $(\alpha h v)^{1/2} \sim h v$ variations of glass samples in (a) Batch I, (b) Batch II.

Optical band gaps for direct and indirect transitions obtained according to the linear regions of the curves in Figures 5(a)-(b) and Figures 6(a)-(b) are as seen in Table 3. Optical band gaps for direct transitions of the samples in Batch I vary between 3.025 eV-3.207 eV and vary between 2.605 eV-2.807 eV for indirect transitions. Optical band gaps for direct transitions of the samples in Batch II vary between 2.963 eV-3.228 eV, and vary between 2.592 eV-2.762 eV for indirect transitions. However, in a similar study that does not contain vanadium, optical band gap and Urbach energy varied between 3.142-2.845 eV and 0.212-0.324 eV, respectively [4]. Binary and ternary borate glasses were prepared with different compositions, in compositions containing zinc or zinc and vanadium, optical band gaps were observed to vary between 2.92-1.14 eV [25].

In both type of transitions, increasing the V_2O_5 ratio in glass compositions led to decrease in optical band gaps. Cu^{2+} doping of undoped group in Batch I glass samples led to the decrease of optical band gaps for direct transitions in the presence of 1% V_2O_5 and led to the

increase of optical band gap in the presence of 0.5% V_2O_5 . However, in indirect transitions, Cu^{2+} doping in both vanadium ratios narrowed the optical band gap. In Batch II glass samples, Cu²⁺ doping in the presence of 1% V₂O₅ and 0.5% V₂O₅ increased the optical band gap. On the contrary, in indirect transitions, Cu²⁺ doping in the presence of 1% V_2O_5 and 0.5% V_2O_5 narrowed the optical band gap. In addition, in Cu² undoped glass compositions, the increase in B2O3/ Na₂O ratio caused narrowing of optical band gaps in both direct and indirect transitions and increase in Urbach energy. Increase in V₂O₅ ratios led to the increase of Urbach energies in Cu^{2+} undoped glasses and decrease in Cu2+ doped glasses. The lowest band gap for direct transitions was observed in glass coded BNAVII-1, the highest band gap was observed in BNAVCuII-2 glass. For indirect transitions, the lowest band gap was observed in BNAVCuII-1 glass and the highest band gap was calculated in BNAVI-2 glass.

Table 3. Thicknesses (d), direct and indirect optical band gaps, Urbach energies of glass samples in two groups.

| Batch I | | | | | |
|------------|--------|------------------|--------------------|---------|--|
| | d (mm) | E _{opt} | Eopt | ΔE (eV) | |
| Glass Code | | (direct) (eV) | (indirect) (eV) | | |
| BNAVI-1 | 2.89 | 3.075 | 2.766 | 0.185 | |
| BNAVI-2 | 2.95 | 3.129 | 2.807 | 0.176 | |
| BNAVCuI-1 | 2.39 | 3.025 | 2.605 | 0.257 | |
| BNAVCuI-2 | 2.83 | 3.207 | 2.730 | 0.308 | |
| Batch II | | | | | |
| | d (mm) | E _{opt} | E _{opt} | ΔE (eV) | |
| Glass Code | | (direct) (eV) | (indirect) (eV) | | |
| BNAVII-1 | 2.64 | 2.963 | 2.634 | 0.209 | |
| BNAVII-2 | 2.58 | 3.113 | 2.762 | 0.206 | |
| BNAVCuII-1 | 2.70 | 3.042 | 2.592 | 0.266 | |
| BNAVCuII-2 | 2.70 | 3.228 | 2.634 | 0.338 | |



Figure 7. $\ln \alpha \sim hv$ variation of glass samples in (a) Batch I, (b) Batch II for Urbach energies.

4. CONCLUSION

Each one of the eight glass samples, prepared in different compositions, has pure amorphous structure, is far from being crystalline and has approximately homogenous distribution. Particularly, decreasing Na2O and Cu²⁺ amount in glass systems minimize the interaction with air, which usually occurs in borate glasses. In the B₂O₃· Na₂O ·Al₂O₃·V₂O₅(:CuO) glass system, increase in V₂O₅ ratio does not only lead to a significant coloring, but shifts the absorption edge to long wavelengths as well. Particularly in glass samples Cu^{2+} , with the increasing containing Na₂O, transmittance maximum shifts towards the long wavelength. Optical band gaps calculated by using the absorption spectra for direct and indirect transitions depend on the ratio of V₂O₅ in the glass composition and the B₂O₃ / Na₂O variation in the structure of the prepared glass.

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