Electron Paramagnetic Resonance Studies of Cu\(^{2+}\) and VO\(^{2+}\) Spin Probes in RO-Li\(_2\)O-Na\(_2\)O-K\(_2\)O-B\(_2\)O\(_3\) (R = Zn, Mg, Sr and Ba) Glass Systems

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Received September 9th, 2011; revised October 13th, 2011; accepted October 23rd, 2011

ABSTRACT

Mixed alkali and alkaline earth oxide glasses of RO-Li\(_2\)O-Na\(_2\)O-K\(_2\)O-B\(_2\)O\(_3\) (R = Zn, Mg, Sr and Ba) system were studied by electron paramagnetic resonance (EPR) and optical absorption spectroscopy. Cu\(^{2+}\) and VO\(^{2+}\) ions were used as the spin probes. The glasses containing 1 mole% of Cu\(^{2+}\) and 2 mole% of VO\(^{2+}\) were prepared by the melt quenching method. X-ray diffraction studies of the samples did not reveal crystalline phases. EPR measurements were made at X-band frequencies with 100 kHz field modulation, at 310 K. Optical spectra were recorded in the wavelength range 300 nm - 800 nm. From the EPR spectra the spin-Hamiltonian parameters were evaluated. The spin-Hamiltonian parameter values in the case of Cu\(^{2+}\) indicated that the ground state of Cu\(^{2+}\) was \(2\)\(^{2}\)\(\text{E}_{\text{g}}\) orbital (\(2\)\(\text{B}_{1g}\)) and the site symmetry around Cu\(^{2+}\) is tetragonally distorted octahedral. The variation of \(g_{\parallel}\) and \(A_{\parallel}\) with the alkaline earth oxide (RO) composition was found to be non-linear which may be due to the change in the ligand field strength at the site of Cu\(^{2+}\) ions. From the spin-Hamiltonian parameters of VO\(^{2+}\), it was observed that the vanadyl ions exist as VO\(^{2+}\) ions in octahedral coordination with tetragonal compression and have C\(_{4V}\) symmetry with ground state \(d_{\text{xy}}\). Tetragonality (\(\Delta g_{\parallel}/\Delta g_{\perp}\)) of V\(^{4+}\) ion sites exhibited non-linear variation with RO content, which indicated change in the ligand field at the site of V\(^{4+}\). A broad optical absorption band was observed in the glass containing Cu\(^{2+}\) ions corresponding to the \(2\)\(^{2}\)\(\text{E}_{\text{g}}\) \(\rightarrow\) \(2\)\(^{2}\)\(\text{B}_{2g}\) transition. From the EPR and optical data the bond parameters were evaluated. In the case of VO\(^{2+}\) ions, the covalency rates were estimated.

Keywords: Borate Glasses; Electron Paramagnetic Resonance (EPR); Optical Absorption; Spin-Hamiltonian Parameters

1. Introduction

Electron Paramagnetic Resonance (EPR) spectroscopy is an experimental technique to obtain information on some of the structural and dynamic phenomenon of a material and to identify the site symmetry around the transition metal (TM) ions in glasses. Glasses doped with TM ions have attracted a great deal of attention because of their potential applications in the development of new tunable solid-state lasers, solar energy converters. It has been well established [1-5] that alkali borate glasses can be used as solid electrolytes, in solid state batteries.

Electrical conductivity, EPR and optical studies were carried on double alkali (mixed alkali) borate glasses by several workers [6-8]. However less work is carried out on glasses containing three alkali oxides [9]. Therefore, in this paper we report the EPR and optical absorption studies on ternary alkali oxide glasses containing alkali earth oxides (ZnO, MgO, SrO, and BaO) using Cu\(^{2+}\) and VO\(^{2+}\) as the spin probes. It is interesting to study the effect of the two paramagnetic probes (Cu\(^{2+}\) and VO\(^{2+}\)) on the structural aspects of the glasses.

2. Experimental

The starting materials used in the present study were analytical grade lithium carbonate (Li\(_2\)CO\(_3\)), sodium carbonate (Na\(_2\)CO\(_3\)), potassium carbonate (K\(_2\)CO\(_3\)), zinc oxide (ZnO), magnesium oxide (MgO), strontium oxide (SrO), barium oxide (BaO) and boric acid (H\(_3\)BO\(_3\)). These materials were weighed to get the required composition and grounded in a mortar with pestle for half an hour to obtain homogeneous mixtures.

The base glass compositions taken were 1) 10RO-10...
Li₂O-10Na₂O-10K₂O-59B₂O₃-1CuO and 2)10RO-10Li₂O-10Na₂O-10K₂O-58B₂O₃-2V₂O₅ (where R = Zn, Mg, Sr and Ba). Each batch was melted in a porcelain crucible in an electric furnace at 1173 K for about 30 minutes. The homogeneous melt was rapidly quenched on to a stainless steel plates maintained at temperature of 373 K. The glasses were annealed for 24 hours at 373 K to relieve the mechanical stresses. For all the glass samples X-ray diffractograms (XRD) were recorded. The featureless, peak free XRD spectra revealed the glassy nature of the samples prepared. Table 1 presents the different glass compositions prepared for the present investigation.

EPR spectra were recorded on dry and perfectly powdered glass samples at room temperature (310 K) using EPR spectrometer (JEOL FEIX) operating at X-band frequency (9.205 GHz) with a modulation frequency of 100 kHz. Uncertainty in the measurement of “g” and “A” were about ±0.002 and ±2 × 10⁻⁴ cm⁻¹ respectively.

The optical absorption spectra of the glasses containing copper were recorded at room temperature (310 K) using UV-VIS spectrophotometer (Shimadzu) in the wavelength region 300 nm to 1100 nm. The uncertainty in the measurement was about ±1 nm. However no optical absorption spectra were observed for the vanadyl glasses.

3. Results and Discussion

3.1. EPR Spectra of Cu²⁺ Ions

The EPR spectra of Cu²⁺ in 10RO-10Li₂O-10Na₂O-10K₂O-59B₂O₃-1CuO are shown in Figure 1. The Cu²⁺ ion, with effective spin S = 1/2, has a nuclear spin I = 3/2 for both ⁶³Cu (natural abundance 69%) and ⁶⁵Cu (natural abundance 31%). Hence, (2I + 1) i.e. four parallel and four perpendicular hyperfine (hf) components were expected. In the present work, three weak parallel components were observed in the lower field region and fourth parallel component was overlapped with the perpendicular component. The perpendicular components in the high field region were not resolved. It was found that high field side of the spectra was more intense than the low field side. The EPR spectra of all the glass samples containing Cu²⁺ ions is similar to those reported for Cu²⁺ ions in other glass systems [7,10-15]. An axial spin-Hamiltonian was employed in the analysis of EPR spectra [16,17] which is given as

\[
H = \beta \left[ g_// \mathbf{H} \cdot \mathbf{S} + g_\perp \left( S_x \mathbf{H}_x + S_y \mathbf{H}_y \right) \right] \\
+ A \left( I_z S_z + I_\gamma S_\gamma \right)
\]

where \(z\) is the symmetry axis, \(\beta\) the Bohr magneton, \(S\) and \(I\) the electron and nuclear spin operators, \(\mathbf{H}_x\), \(\mathbf{H}_y\) and \(\mathbf{H}_z\) the static magnetic field components, \(g_//\) and \(g_\perp\) the parallel and perpendicular components of “g” tensor.

Table 1. Spin-Hamiltonian parameters, optical absorption bands and bonding parameters of Cu²⁺ ions in the glass systems.

<table>
<thead>
<tr>
<th>Glass system (Glass Code)</th>
<th>(g_//)</th>
<th>(g_\perp)</th>
<th>(A_// \times 10^4) (cm⁻¹)</th>
<th>(A_\perp \times 10^4) (cm⁻¹)</th>
<th>(\Delta E_{yy}) (cm⁻¹)</th>
<th>(\alpha^2)</th>
<th>(\beta^2)</th>
<th>(\beta_1^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10ZnO-10Li₂O-10Na₂O-10K₂O-59B₂O₃-1CuO (ZLNKBC)</td>
<td>2.266</td>
<td>2.030</td>
<td>145.03</td>
<td>25.32</td>
<td>13333.3</td>
<td>0.726</td>
<td>0.818</td>
<td>0.734</td>
</tr>
<tr>
<td>10MgO-10Li₂O-10Na₂O-10K₂O-59B₂O₃-1CuO (MLNKBC)</td>
<td>2.265</td>
<td>2.045</td>
<td>138.88</td>
<td>27.77</td>
<td>13333.3</td>
<td>0.709</td>
<td>0.729</td>
<td>0.651</td>
</tr>
<tr>
<td>10SrO-10Li₂O-10Na₂O-10K₂O-59B₂O₃-1CuO (SLNKBC)</td>
<td>2.254</td>
<td>2.036</td>
<td>138.88</td>
<td>27.77</td>
<td>13698.3</td>
<td>0.696</td>
<td>0.849</td>
<td>0.747</td>
</tr>
<tr>
<td>10BaO-10Li₂O-10Na₂O-10K₂O-59B₂O₃-1CuO (BLNKBC)</td>
<td>2.253</td>
<td>2.031</td>
<td>139.27</td>
<td>41.78</td>
<td>12987.0</td>
<td>0.693</td>
<td>0.852</td>
<td>0.708</td>
</tr>
</tbody>
</table>
while $A_{//}$ and $A_{\perp}$ are parallel and perpendicular components of the hyperfine tensor $A$. The nuclear quadrupole contribution is neglected [18].

The solution to the spin-Hamiltonian gives the following expressions for the peak position related to the principal values of $g$ and $A$ tensors [19], for the parallel and perpendicular hyperfine peaks respectively.

$$h\nu = g_{//}\beta H + m A_{//} + \left(\frac{15}{4} - m^2\right) A_{//}^2 \frac{A_{//}^2}{2g_{//}\beta H}$$

(2)

and

$$h\nu = g_{\perp}\beta H + m A_{\perp} + \left(\frac{15}{4} - m^2\right) A_{\perp}^2 + A_{\perp}^2 \frac{A_{\perp}^2}{4g_{\perp}\beta H}$$

(3)

Here $m$ is the nuclear magnetic quantum number of the copper nucleus with the values $+3/2$, $+1/2$, $-1/2$ and $-3/2$ and $\nu$ is the microwave frequency. The spin-Hamiltonian parameters have been evaluated and are presented in Table 1.

It was observed that, $g_{//} > g_{\perp} > g_{e} = 2.0023$. From the "$g$" values and the shape of the EPR spectra it can be concluded that the ground state of Cu²⁺ ions is $d^{9}$ orbital ($^{2}B_{1g}$ state), the Cu²⁺ ions being located in tetragonally distorted octahedral sites [20-24]. The high "$g$" values indicate the presence of a CuO₆ chromophore [25, 26]. The line width of the parallel hyperfine components was found to increase with increasing values of the nuclear spin quantum number $m$ (Figure 1), which may be due to fluctuation in both the ligand fields and bond covalencies from one copper(II) complex to the next, giving rise to a narrow distribution in $g$ [24,27]. It can be observed from Figure 2 that the variation of $g_{//}$ and $A_{//}$ for different ZnO, MgO, SrO and BaO composition is non-linear. This may be due to change in the tetragonal distortion. Variation in $g$ and $A$ values may be associated with the change in the environment of Cu²⁺, i.e. in the ligand field strength at the site of Cu²⁺ which may be attributed to the structural changes in the glass. In the B₂O₃ glasses the addition of the network modifiers will lead to an increase in the coordination number of some portion of the boron atoms from 3 to 4. The resulting glass may be composed of both triangular and tetrahedral units which form a relatively open network with holes between the oxygen atoms of sufficient size to accommodate the alkali and alkaline earth oxides [28]. As a doubly charged cation, R²⁺ is sufficiently strong to split the network. Therefore, sufficient non-bridging oxygen’s will be available for coordination in the broken network. The alkali oxides make available additional weakly bonded O²⁻ for each R²⁺, i.e. R²⁺ captures the O²⁻ from alkali oxide which happens at the expense of alkali oxide coordination. The solubility of the Cu²⁺ ions increases with

3.1.1. Optical Absorption Spectra

The optical absorption spectra of all the glasses containing Cu²⁺ ions resulted in a broad absorption band. The observed peak positions of the optical absorption spectra of the glasses are listed in Table 1. The observed broad band was assigned to the $^{2}B_{1g} \rightarrow ^{2}B_{2g}$ transition of Cu²⁺ ions [12]. The variation of peak position of the optical absorption band with composition (ZnO, MgO, SrO and BaO) is shown in Figure 3. The variation is found to be non-linear. It is observed that SLNKBC glass has the lowest value of absorption peak wavelength, which may be due to increase in the ligand field strength around

![Figure 2. Variation of $g_{//}$ and $A_{//}$ with RO composition.](image)

![Figure 3. Variation of optical absorption maximum ($\lambda_{\text{max}}$) with RO composition.](image)
Cu$^{2+}$ ion. The optical absorption spectrum is influenced by the host structure into which the TM ions are incorporated. In oxide glasses, the TM ions mostly form coordination complexes with doubly charged oxygen as the ligands. However Cu$^{2+}$, being as d$^9$ ion, experiences a moderate covalency for the in-plane $\sigma$- and $\pi$-bonding respectively while the $\beta^2$ value indicated that the out-of-plane $\pi$-bonding is slightly ionic in nature.

3.2. EPR Spectra of VO$^{2+}$ Ions

The EPR Spectra of VO$^{2+}$ ions in 10RO-10Li$_2$O-10Na$_2$O-10K$_2$O-58B$_2$O$_3$-2V$_2$O$_5$ are shown in Figure 4. The spectra have structures that are characteristic of hyperfine interactions arising from an unpaired electron with $^{51}$V nucleus, whose spin is 1/2 and present in 99.75% abundance. These spectra were analyzed by assuming [45-47] that vanadium is present as vanadyl ion in a ligand field of C$_{4v}$ symmetry. The EPR spectra were analyzed by using an axial spin-Hamiltonian (Equation (1)). The solutions of the spin-Hamiltonian [20], for parallel and perpendicular hyperfine lines are given respectively as:

$$H_{||} (m) = H_{||} (0) - mA_{||} - \frac{63}{4} - m^2 \frac{A_1^2}{2H_{||} (0)}$$  (8)

$$H_{\perp} (m) = H_{\perp} (0) - mA_{\perp} - \frac{63}{4} - m^2 \frac{A_1^2 + A_2^2}{4H_{\perp} (0)}$$  (9)

where $m$ is the magnetic quantum number of the vanadium nucleus having the values of $\pm 7/2, \pm 5/2, \pm 3/2, \pm 1/2$, $H(0) = (h v g_{\perp} \beta)$ and $H_{||} (0) = (h v g_{||} \beta)$. The spin-Hamiltonian parameters for various compositions were calculated using Equations (8) and (9) and are listed in Table 2. The paramagnetism of the vanadyl ion (V$^{4+}$) arises from a single unpaired electron, as the crystalline fields quench the orbital angular momentum. The crystal fields of V$^{4+}$ ions in glasses can be described either by threefold or fourfold symmetries [48]. The variation of $g_{\perp}$ and $g_{||}$ depend critically on the local symmetry of this field. Although the V$^{4+}$ ion usually in six-fold coordination in complexes containing vanadyl, its local symmetry is generally a distorted octahedron of oxygen ions.

An octahedral site with tetragonal compression would give values of $g_{\perp} < g_{||} < g_{e}$. In the present investigation, it is observed that of $g_{||} < g_{\perp} < g_{e}$ and $A_{||} > A_{\perp}$. It is therefore concluded that V$^{4+}$ in the present glass samples exist as VO$^{2+}$ ions in octahedral coordination with tetragonal compression. The symmetry of vanadyl complex is C$_{4v}$, and the ground state of 3d$^1$ ion is $d_{xy}$. The measure of tetragonality of the VO$^{2+}$ site is given by $\Delta g_{\perp}/\Delta g_{||} \left[ \Delta g_{\perp} = g_{\perp} - g_{||} \text{ and } \Delta g_{||} = g_{||} - g_{e} \right]$ values (Table 3). The glass (ZLNKBV) has high $\Delta g_{\perp}/\Delta g_{||}$ value compared to other glasses (MLNKBV, SLNKBV, and BLNKBV). The high (Ag$^1$/Ag$^2$) value for (ZLNKBV) glass indicates that the vanadyl ions in the glass (ZLNKBV) are more tetragonally distorted. The low value of Ag$^1$ for the glass (ZLNKBV) also support that vanadyl ions in this glass
Table 2. Spin-Hamiltonian parameters of VO$^{2+}$ ion in the glass systems.

<table>
<thead>
<tr>
<th>Glass system (Glass code)</th>
<th>$g_{\parallel}$</th>
<th>$g_{\perp}$</th>
<th>$\Delta g_{\parallel}$</th>
<th>$\Delta g_{\perp}$</th>
<th>$A_{\parallel} \times 10^4$ (cm$^{-1}$)</th>
<th>$A_{\perp} \times 10^4$ (cm$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZnO MgO Sr2O BaO</td>
<td>1.941</td>
<td>1.996</td>
<td>0.0613</td>
<td>0.0063</td>
<td>181.50</td>
<td>67.03</td>
</tr>
<tr>
<td>ZnO MgO Sr2O BaO</td>
<td>1.957</td>
<td>1.983</td>
<td>0.0453</td>
<td>0.0193</td>
<td>173.16</td>
<td>75.41</td>
</tr>
<tr>
<td>ZnO MgO Sr2O BaO</td>
<td>1.944</td>
<td>1.976</td>
<td>0.0583</td>
<td>0.0263</td>
<td>178.75</td>
<td>83.79</td>
</tr>
<tr>
<td>ZnO MgO Sr2O BaO</td>
<td>1.950</td>
<td>1.978</td>
<td>0.0523</td>
<td>0.0243</td>
<td>175.95</td>
<td>78.20</td>
</tr>
</tbody>
</table>

Figure 4. EPR spectra of VO$^{2+}$ ions in RO-Li$_2$O-Na$_2$O-K$_2$O-B$_2$O$_3$ glasses.

are more tetragonally distorted. Figure 5 shows the variation of $\Delta g_{\parallel}/\Delta g_{\perp}$ with different compositions (ZnO, MgO, SrO, and BaO). The variation is non-linear which may due to change of ligand field at the transition metal (TM) ion site. For the glasses containing (MgO, SrO, BaO) the ($\Delta g_{\parallel}/\Delta g_{\perp}$) values decrease in the order (MgO, SrO, BaO) as shown in Table 3. The decrease in the values $\Delta g_{\parallel}/\Delta g_{\perp}$ suggests that the octahedral symmetry in these glasses is improved [49].

The Fermi contact interaction term $K$, the dipolar hyperfine coupling parameter $P$ and the covalency rates ($1 - \alpha^2$) and ($1 - \nu^2$) were calculated using the following equations [50].

The parameter $\beta^2$ which is measure of out-of-plane $\pi$-bonding with the equitorial ligands is assumed to be unity for many oxide glasses [50]. ($1 - \alpha^2$) and ($1 - \nu^2$) represent the covalency rates. ($1 - \nu^2$) provides an estimate of covalency of the $\pi$-bonding between the V$^{4+}$ ion and the vanadyl oxygen, while ($1 - \alpha^2$) gives an estimation of the $\sigma$-bonding with the equatorial ligands. The covalency rates were estimated by taking the values of $\Delta E_1 = 12500$ cm$^{-1}$ and $\Delta E_2 = 16000$ cm$^{-1}$ [51]. The spin orbit coupling constant ($\lambda$) is taken as 249 cm$^{-1}$ [50-52]. The covalency rates ($1 - \alpha^2$) and ($1 - \nu^2$) are given in Table 3. The values of ($1 - \alpha^2$) and ($1 - \nu^2$) indicate a moderate covalency for $\sigma$ and $\pi$-bonds. These values indicate only the trends in the variation of magnitude of bonding parameters. The Equations (10) and (11) can be
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### Table 3. Tetragonality and covalency rates of V\(^{4+}\) ion.

<table>
<thead>
<tr>
<th>Glass System</th>
<th>(\Delta g_{|}/\Delta g_{\perp})</th>
<th>(\Delta \xi \times 10^4) (cm(^{-1}))</th>
<th>(\Delta \eta \times 10^4) (cm(^{-1}))</th>
<th>((1-\alpha^2))</th>
<th>((1-\nu^2))</th>
<th>(P \times 10^6) (cm(^{-1}))</th>
<th>(K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZLNKBV</td>
<td>9.73</td>
<td>63.41</td>
<td>51.06</td>
<td>0.507</td>
<td>0.839</td>
<td>124.90</td>
<td>0.945</td>
</tr>
<tr>
<td>MLNKBV</td>
<td>2.35</td>
<td>56.58</td>
<td>41.17</td>
<td>0.635</td>
<td>0.513</td>
<td>109.15</td>
<td>1.068</td>
</tr>
<tr>
<td>SLNKBV</td>
<td>2.22</td>
<td>52.60</td>
<td>42.36</td>
<td>0.531</td>
<td>0.337</td>
<td>104.80</td>
<td>1.204</td>
</tr>
<tr>
<td>BLNKBV</td>
<td>2.15</td>
<td>55.19</td>
<td>42.56</td>
<td>0.579</td>
<td>0.387</td>
<td>108.51</td>
<td>1.113</td>
</tr>
</tbody>
</table>

rewritten as,

\[
A_{\perp} = -P\left[\left(\frac{4}{7}\right)\beta_K^2 + \Delta g_{\perp} + \left(\frac{3}{7}\right)\Delta g_{\|}\right] - P\beta_K^2 K
\]

\[
A_{\|} = P\left[\left(\frac{2}{7}\right)\beta_K^2 - \left(\frac{11}{14}\right)\Delta g_{\perp}\right] - P\beta_K^2 K
\]

From the molecular orbital theory, it was observed [50] that the components \(A_{\|}\) and \(A_{\perp}\) consist of the contributions \(A_{\|}\) and \(A_{\perp}\) of the 3\(d_{xy}\) electron to the hyperfine structure. \(P\beta_K^2 K\) term arises due to the anomalous contribution of the s-electrons. The values of \(P, K, A_{\|}\) and \(A_{\perp}\) were calculated and are given in Table 3.

The decrease of the anisotropic contribution \(A_{\|}\) and \(A_{\perp}\) of the 3\(d_{xy}\) electron to the hyperfine splitting for the glasses containing MgO, BaO and SrO is brought about by the increase of screening of the 3\(d_{xy}\) orbital from its nucleus through the overlap of the electron orbits of the surrounding ligands of oxygen [49]. This is also supported by the decrease in the value of \(P\), and increase in the value of \(K\). The high values of \(K\) indicate a large contribution to the hyperfine constant by the “s” electron.

### 4. Conclusions

The EPR studies revealed that the Cu\(^{2+}\) ions are present in all the glass systems investigated and they exist in tetragonally distorted octahedral sites with \(d_{x^2-y^2}\) (B\(_{1g}\)) ground state. The spin-Hamiltonian parameters are influenced by the change in the glass composition. The bond parameter values indicated that in plane \(\sigma\) and \(\pi\)-bodings are moderately covalent. The out-of-plane \(\pi\)-bonding is slightly ionic in nature.

In all the glass systems, the vanadium ions (V\(^{4+}\)) exist as VO\(^{2+}\) ions in octahedral coordination with tetragonal compression and belong to C\(_{4v}\) symmetry. The ground state of VO\(^{2+}\) is \(d_{xy}\). The variation of \(\Delta g_{\|}/\Delta g_{\perp}\) with different compositions (ZnO, MgO, SrO and BaO) is non-linear which may be due to change of electric field at the transition metal (TM) ion site. The \((1-\alpha^2)\) and \((1-\nu^2)\) values indicated moderate covalency for \(\sigma\) and \(\pi\)-bonds respectively.

### REFERENCES


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