

Research Article

Structural and Physical Properties of $\text{Fe}_2\text{O}_3\text{-B}_2\text{O}_3\text{-V}_2\text{O}_5$ Glasses

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The structural and physical properties of $x\text{Fe}_2\text{O}_3\text{-(40-x) B}_2\text{O}_3\text{-60V}_2\text{O}_5$ ($0 \leq x \leq 20$) glass system have been investigated. The samples were prepared by normal melt-quench technique. The structural changes were inferred by means of FTIR by monitoring the infrared (IR) spectra in the spectral range $600\text{--}4000\text{ cm}^{-1}$. The absence of boroxol ring (806 cm^{-1}) in the present glass system suggested that these glasses consist of randomly connected BO_3 and BO_4 units. The conversion of BO_3 to BO_4 and VO_5 to VO_4 tetrahedra along with the formation of non-bridging oxygen's (NBOs) attached to boron and vanadium takes place in the glasses under investigation. The density and molar volume of the present glass system were found to depend on Fe_2O_3 content. DC conductivity of the glass system has been determined in the temperature range $310\text{--}500\text{ K}$. It was found that the general behavior of electrical conductivity was similar for all glass compositions and found to increase with increasing iron content. The parameters such as activation energy, average separation between transition metal ions (TMIs), polaron radius, and so forth have been calculated in adiabatic region and are found consistent with Mott's model of phonon-assisted polaronic hopping.

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1. Introduction

Borate glasses are generally insulating in nature, and the addition of transition metal oxide such as Fe_2O_3 and V_2O_5 makes these glasses semiconducting [1, 2]. These semiconducting glasses have been extensively studied owing to their potential applications as optical and electrical memory switchings, cathode materials for making solid state devices, and optical fiber [3–5].

Structure of the borate glasses has been studied by various physical and chemical methods including Raman and Infrared spectroscopy, Brillouin experiments, NMR, and neutron scattering investigations [6–9]. Infrared (IR) spectroscopy is one of the important techniques which are used to study the local arrangement in inorganic glasses. In oxide glasses, B_2O_3 is a basic glass former because of its higher bond strength, lower cation size, smaller heat of fusion, and trivalency of boron. In these glasses, boron (B^{3+}) ions are triangularly coordinated by oxygen to form glasses easily. The main structural units of vitreous B_2O_3 glasses are BO_3 triangles forming three member (boroxol) rings

connected by B–O–B linkage [10]. Boroxol ring is a high-planar ring with a bond length $1.36 \pm 0.005\text{ \AA}$, whereas the B–O bond length for BO_4 tetrahedra was observed to be $1.47 \pm 0.01\text{ \AA}$ [11]. In IR spectra of borate glasses, the boroxol ring has its characteristic absorption at 806 cm^{-1} [12]. It has been observed that the structure of these glasses depends on the nature of the network formers as well as the network modifier. It has been reported that addition of a network modifier in borate glasses could produce the conversion of the triangular BO_3 structural units to BO_4 tetrahedra with coordination number of 4, which are incorporated in more complex cyclic groups such as diborate, triborate, tetra or pentaborate, and the formation of NBO atoms [8, 9].

Transition-metal oxides (TMOs) glasses exhibit semiconducting properties due to the existence of TMIs in more than one valence state [1]. The electron-phonon interaction in these glasses is strong enough to form small polaron, and the electrical conduction process occurs by the hopping of small polarons between different valence states as proposed by Austin and Mott [2]. Hopping conduction in these glasses was generally known to be adiabatic for V_2O_5 content

above 50 mol% [13], and for $V_2O_5 < 50$ mol%, conduction becomes non-adiabatic [14].

Sufficient work regarding the structural and physical properties of different oxide glasses having TeO_2 and P_2O_5 as glass former has been reported [15–18], but relatively few work has been done on semiconducting oxide glasses (having Fe and V as TMIs) with B_2O_3 as a network former. The objective of present paper is to study the structural and physical properties of iron-boro-vandate glasses to shed some light on the role of the Fe_2O_3 in this glass system.

2. Experimental Details

2.1. Sample Preparation

Iron-doped vanadium borate glasses were prepared from analytical reagent grade powder of Fe_2O_3 , V_2O_5 , and H_3BO_3 of high purity which are thoroughly mixed, in appropriate proportions. The batch materials were dry mixed and melted in porcelain crucibles placed in an electrically heated muffle furnace at 1473 K for about two hours, until a bubble-free liquid was formed. The melts were quickly cooled at room temperature by pouring and pressing between two stainless plates. As obtained glass samples were polished and finally cut into desired size (≈ 10 mm \times 10 mm \times 1 mm).

2.2. FTIR Measurements

The vibration spectra of the glass system were obtained at room temperature using FTIR spectrophotometer model ABB Bomen (MB-Series) in the range 600–4000 cm^{-1} . The measurements were performed directly on glass pallets obtained as above.

2.3. Density Measurements

The density “ d ” of the glasses was determined at room temperature using Archimedes principle with Xylene as an inert immersion liquid.

The molar volume V_M of each glass sample was calculated using formula [19]

$$V_M = \sum \frac{x_i M_i}{d}, \quad (1)$$

where x_i is the molar fraction, and M_i is the molecular weight of the i th component.

2.4. D.C. Conductivity Measurements

The conductivity measurements were carried out by using Keithly electrometer (Model 617) in the temperature range of 310–500 K. Silver paste electrodes were deposited on both faces of the polished samples. The absence of the barrier layers at the contacts was confirmed by linear $I - V$ characteristics.

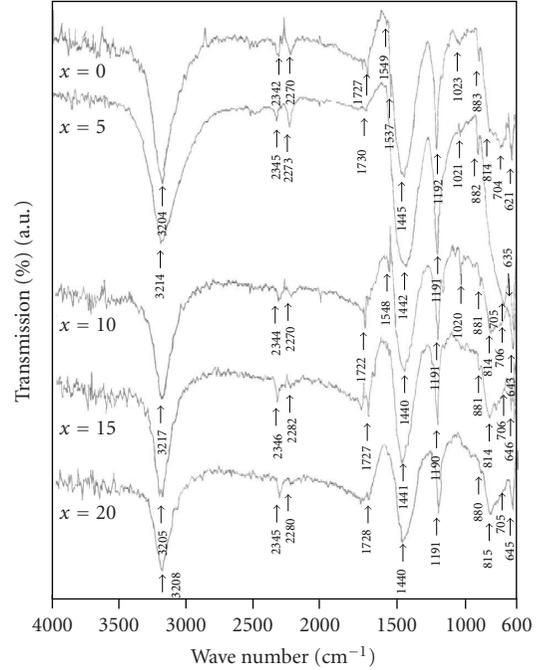


FIGURE 1: IR transmission spectra of $xFe_2O_3-(40-x) B_2O_3-60V_2O_5$ glasses.

3. Results and Discussion

3.1. FTIR Analysis

The infrared spectra of $xFe_2O_3-(40-x) B_2O_3-60V_2O_5$ glasses, with $x = 0, 5, 10, 15,$ and 20 mol%, are shown in Figure 1. The vibrational modes of the borate network are seen to be mainly active in three infrared regions which are similar to those reported earlier [20, 21]. The group of bands that occur at 1200–1600 cm^{-1} is due to the asymmetric stretching relaxation of the B–O band of trigonal BO_3 units. The second group of band lies between 800–1200 cm^{-1} and is due to the B–O bond stretching of the tetrahedral BO_4 units. The third group of absorption bands is observed around 700 cm^{-1} and is due to bending of B–O–B linkages in the borate network. Similar results have been reported for $V_2O_5-B_2O_3$ glasses [22, 23].

The absorption peaks assigned in IR spectra of glasses under study are listed in Table 1. The absorption peak observed in all glass samples at 3204–3217 cm^{-1} is attributed to hydroxol or water groups [24] and it is due to hygroscopic nature of glass samples [25]. The peaks around 2270–2286 cm^{-1} and 2342–2345 cm^{-1} are attributed to –OH group [26]. The absorption peak around 1722–1730 is due to H–O–H bending [22]. In the present glass system, the absence of peak at 806 cm^{-1} indicates the absence of boroxol ring formation [12], which suggests that the glass system under investigation consists of randomly connected BO_3 and BO_4 groups. The absorption peaks at 1192, 1445, 1537, and 1549 cm^{-1} [27, 28] are related with the fundamental asymmetrical stretching vibration of the B–O bond of the trigonal BO_3 units. The absorption bands observed at 814, 883, and 1023 cm^{-1} [20, 29] are assigned to the stretching

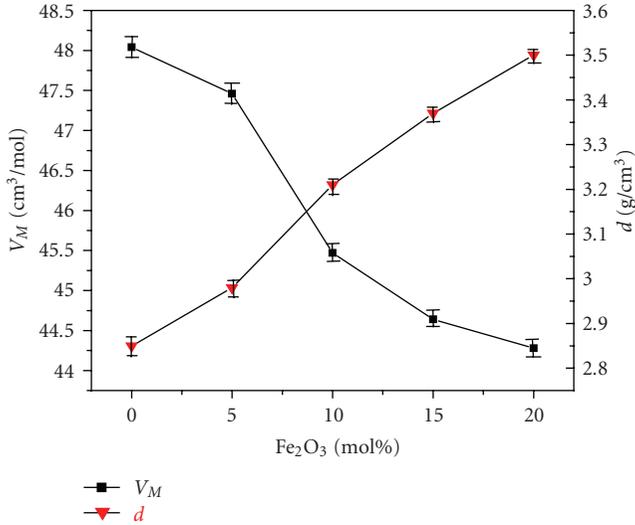


FIGURE 2: Variation of density and molar volume with Fe_2O_3 content.

vibrations of B–O bond of tetrahedral BO_4 units and are shifted toward lower wave numbers with increasing iron content. It is also observed (Figure 1) that on increasing Fe_2O_3 content, the absorption band at around 1445 cm^{-1} shifts toward lower wave number ($1445\text{--}1440\text{ cm}^{-1}$) with noticeable decrease in intensity. In the present glass system, the shift of the vibrational band from higher to lower wave number is ascribed to the increase in the bond length of B–O groups and the formation of BO_4 units. The decrease in intensity of vibrational bands (for $x \leq 10\text{ mol}\%$) at 1445 and 1192 cm^{-1} and clear appearance of band in the lower region at 814 cm^{-1} suggest the formation of NBOs. A sharp absorption band observed at around 1192 cm^{-1} may be attributed to triangular B–O stretching vibrations of BO_3 units [27]. On increasing the Fe_2O_3 contents, the frequency of this band remains almost the same, revealing the strong appearance of the triangular borate units (BO_3). The absorption band observed at 621 cm^{-1} is due to the bending of O–B–O linkage [30]. The shifting of this band toward higher wave number ($621\text{--}646\text{ cm}^{-1}$) with increasing Fe_2O_3 content indicates the formation of FeO_4 group [30]. This possibility is more in the borate glasses in which boroxol rings are absent. The absorption peak observed at 704 cm^{-1} is assigned to B–O–B bending vibration in borate network [31]. At low concentration of Fe_2O_3 , a weak absorption band observed at 814 cm^{-1} originates from stretching vibration of V–O–V bridges [32] and becomes slight intense as the concentration of Fe_2O_3 increases. The weak absorption band appearing in all the samples at $\approx 883\text{ cm}^{-1}$ is due to V_2O_5 content. The shoulder at 1023 cm^{-1} can be referred to stretching vibrations of BO_4 tetrahedra and is also due to higher content of V_2O_5 in the glass system. It has been reported that the V_2O_5 structure is built up by deformed VO_5 trigonal bonded zigzag chains. Each VO_5 group contains a short V=O bond (Vanadyl group), which shows its characteristic vibration band at around 1023 cm^{-1} [33]. The appearance of this frequency band at 1020 cm^{-1} at $x = 10\text{ (mol}\%)$ and the clear separation

of the peak at 814 cm^{-1} on increasing Fe_2O_3 contents suggest that this absorption is not only due to the presence of non-bridging V–O⁻¹ and V=O bond, but also due to the presence of BO_4 tetrahedra. Similar results have also been observed in potassium-boro-vanadate-iron glasses [34]. From Figure 1, it can be clearly inferred that a weak absorption band corresponding to 814 cm^{-1} appears for low concentration of Fe_2O_3 . This peak, however, disappears as the concentration of Fe_2O_3 increases ($x = 5\text{ mol}\%$) and with further increase in Fe_2O_3 content ($x = 10\text{ mol}\%$), the increase in intensity of this peak indicates that the Fe_2O_3 acts as a glass modifier. The intensity of BO_3 structural units decreases on further increases of Fe_2O_3 contents, and clear appearance of peaks ($\approx 814\text{ cm}^{-1}$) in the lower region suggests that some BO_3 structural units are converted into some BO_4 tetrahedral units, which results in the formation of NBOs. On further addition of Fe_2O_3 contents ($15\text{--}20\text{ mol}\%$), the absorption bands observed in the lower region, that is, at 1190 , 881 , 814 , 706 , and 646 cm^{-1} remain almost unaffected, however, their intensities changes which indicate that Fe_2O_3 also acts as a glass former. These observations suggest that at low concentration ($x \leq 10\text{ mol}\%$), Fe_2O_3 acts as a glass modifier and at high concentration ($x = 15\text{--}20\text{ mol}\%$), Fe_2O_3 acts as a glass former. Therefore, in the present glass system, Fe_2O_3 acts both, as a glass modifier as well as a glass former. The results obtained are well consistent with already reported result [26].

3.2. Density and Molar Volume

The determined values of density “ d ” and molar volume “ V_M ” of the glass samples are presented in Table 3. In general, the density of glass system is explained in terms of a competition between the masses and sizes of the various structural units present in glass. In other words, the density is related to how tightly the ions and ionic groups are packed together in the structure. The variation of density as a function of glass composition (x) is shown in Figure 2. It is observed that the density increases gradually with the increase in Fe_2O_3 content in the present glass system. The relationship between density and glass composition (x) can be explained in terms of an apparent volume V_M occupied by 1 gm atom of oxygen. The value of V_M has been calculated from the density and composition using the formula reported earlier [19], and its composition dependence is shown in Figure 2. It is observed that molar volume decreases monotonically with the increase of Fe_2O_3 content which indicates that the topology of the network is significantly changed with composition. On the other hand, these trends can be explained rather simply due to the replacement of a lighter cation (B) by a heavier one (Fe). As observed in IR spectra, the addition of Fe_2O_3 causes increase in NBOs, which in turn randomizes the structure and, therefore, glass structure becomes relatively more open. These results are found consistent with the results reported earlier [35, 36].

3.3. DC Conductivity

The temperature dependence of dc conductivity “ σ ” for the different glass compositions is shown in Figure 3. It is

TABLE 1: Peak frequencies from IR spectra for $x\text{Fe}_2\text{O}_3-(40-x)\text{B}_2\text{O}_3-60\text{V}_2\text{O}_5$ glass system.

x (mol%)	Peak position (cm^{-1})												
	O–B–O	B–O–B	BO_4			BO_3			OH ⁻¹ group				
0	621	704	814	883	1023	1192	1445	1537	1549	1727	2270	2342	3204
5	635	705	—	882	1021	1191	1442	—	—	1730	2273	2345	3214
10	643	706	814	881	1020	1191	1440	—	1548	1722	2270	2344	3217
15	646	706	814	881	—	1190	1441	—	—	1727	2282	2346	3205
20	645	705	815	880	—	1191	1440	—	—	1728	2286	2345	3208

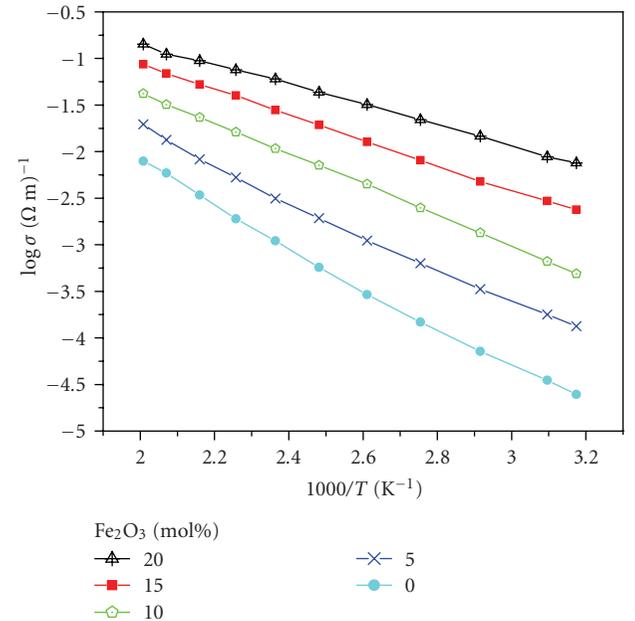
TABLE 2: Electrical conductivity at different temperatures and the prefactor of Mott-Austin formula of $x\text{Fe}_2\text{O}_3-(40-x)\text{B}_2\text{O}_3-60\text{V}_2\text{O}_5$ glasses.

x (mol%)	$\sigma_{320\text{K}} (\Omega\text{m})^{-1}$	$\sigma_{400\text{K}} (\Omega\text{m})^{-1}$	$\sigma_{500\text{K}} (\Omega\text{m})^{-1}$	$\log \sigma_0$
0	0.313×10^{-4}	5.435×10^{-4}	7.945×10^{-3}	2.673
5	1.600×10^{-4}	1.783×10^{-3}	1.987×10^{-2}	2.793
10	5.817×10^{-4}	7.705×10^{-3}	4.339×10^{-2}	1.750
15	2.680×10^{-3}	1.794×10^{-2}	8.853×10^{-2}	1.581
20	8.220×10^{-3}	4.194×10^{-2}	14.73×10^{-2}	1.450

TABLE 3: Chemical composition and physical properties of $x\text{Fe}_2\text{O}_3-(40-x)\text{B}_2\text{O}_3-60\text{V}_2\text{O}_5$ glasses.

x (mol%)	Molar volume V_M (cm^3/mol)	Density d (g/cm^3)	W (eV)
0	48.04	2.85	0.427
5	47.46	2.98	0.361
10	45.47	3.21	0.329
15	44.64	3.37	0.266
20	44.28	3.50	0.220

observed that conductivity increases smoothly with increasing temperature, indicating temperature dependence activation energy “ W ” which is characteristic of small polaron hopping (SPH) conduction mechanism in TMO glasses [37]. As shown in Figure 3, the logarithmic conductivity in the temperature range (310–500 K) exhibits a linear dependence on reciprocal temperature. The composition dependence of dc conductivity at particular temperature (Figure 4) indicates that the conductivity increases with increasing Fe_2O_3 content. The activation energy calculated from the slop of the graphs (Figure 3) is listed in Table 3. It is clear from Figure 5 that the activation energy decreases with increasing Fe_2O_3 content. The composition dependence of dc conductivity at 400 K (Figure 4) and activation energy (Figure 5) indicate that the variation of “ σ ” as well as “ W ” with composition is much faster for the present glass system than those for the traditional vanadate glasses. It is observed that as the concentration of Fe_2O_3 increases, the activation of electrical conduction decreases and electrical conductivity increases. The low value of activation energy and high value of electrical conductivity are similar to those of $\text{V}_2\text{O}_5\text{-BaO-B}_2\text{O}_3$ glasses [38]. The change in conductivity and activation energy may help to detect the structural changes as a consequence of increasing of Fe_2O_3 content and decreasing of boron oxide content. Generally, it is known that addition

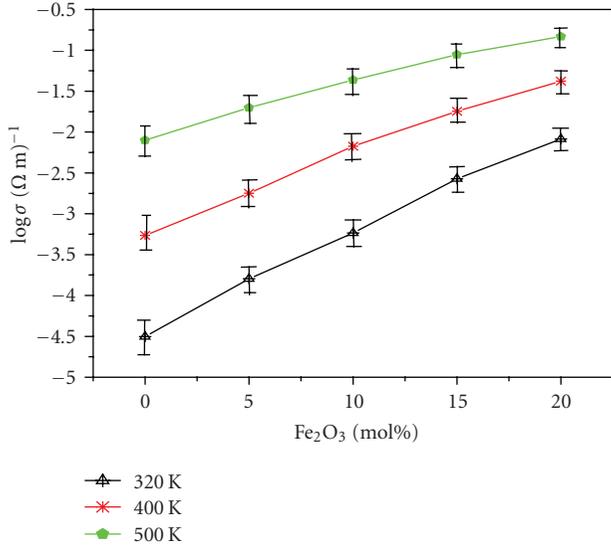
FIGURE 3: Variation of $\log \sigma$ versus $1000/T(\text{K}^{-1})$ for studied glasses.

of Fe_2O_3 in borate glasses increases the conductivity as result of increasing of NBO ions [39]. It is clear from Figures 4 and 5 that the magnitude of conductivity is higher for those compositions which have lower activation energy. This result is in consistent with the small polaron hopping theory [40]. According to this theory, the conduction process at higher temperature is considered in terms of optical phonon assisted hopping of small polaron between localized states. The dc conductivity in adiabatic region is given by

$$\sigma = \sigma_0 \exp\left(-\frac{W}{kT}\right), \quad (2)$$

TABLE 4: Hopping parameters of $x\text{Fe}_2\text{O}_3-(40-x)\text{B}_2\text{O}_3-60\text{V}_2\text{O}_5$ glasses.

x (mol%)	$n_{\text{Fe}_2\text{O}_3}$ (cm^{-3})	$n_{\text{V}_2\text{O}_5}$ (cm^{-3})	N (cm^{-3})	R (nm)	r_p (nm)
0	0×10^{23}	0.1131×10^{23}	0.1131×10^{23}	0.3212	0.179
5	0.0112×10^{23}	0.1183×10^{23}	0.129×10^{23}	0.3074	0.171
10	0.024×10^{23}	0.1274×10^{23}	0.151×10^{23}	0.2917	0.163
15	0.038×10^{23}	0.1337×10^{23}	0.171×10^{23}	0.2799	0.156
20	0.052×10^{23}	0.1389×10^{23}	0.191×10^{23}	0.2697	0.150

FIGURE 4: Effect of Fe_2O_3 content on dc conductivity at different temperatures.

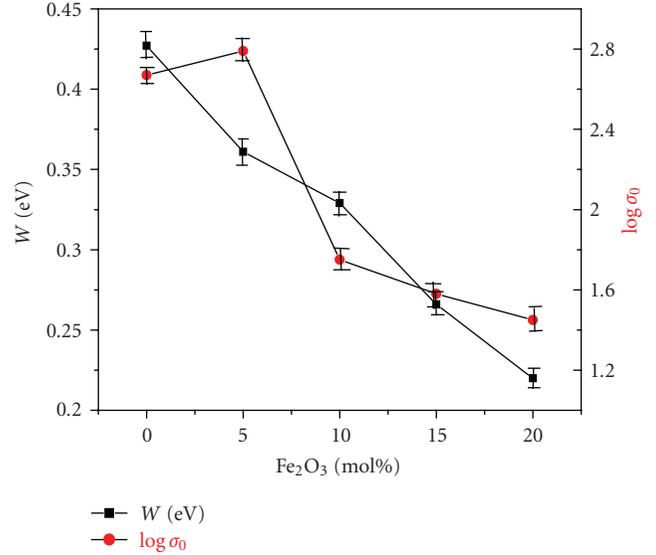
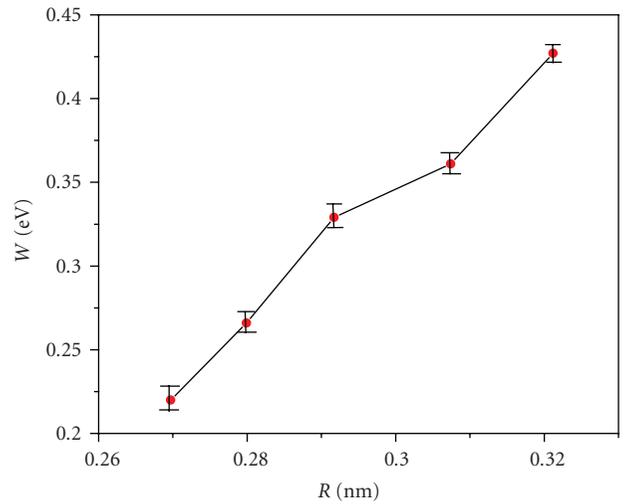
where σ_0 is a pre-exponential factor, W is the activation energy, k is Boltzmann constant, and T is temperature in Kelvin. The values of $\log \sigma_0$ were determined from the intercept of the conductivity versus temperature curve (Figure 3). The observed values of $\log \sigma_0$ are found to be independent of Fe_2O_3 content (Figure 5) which confirms the adiabatic SPH for the present glass system. The present glass system consists of two types of TMIs, that is, Fe and V. The density “ n ” of respective ions was calculated (in order to confirm the relation between the activation energy W and mean distance R) using the formula [14]:

$$n = 2 \left[\left(\frac{dW_t}{M_w} \right) N_A \right], \quad (3)$$

where d , N_A , W_t , and M_w are the measured density of the sample, Avogadro’s number, weight fraction, and molecular weight of the respective ions (Fe & V), respectively. The calculated values of iron and vanadium ions are listed in Table 4. The average distance “ R ” between the transition ions (assuming homogenous distribution of transition ion in glass volume) was calculated as follows:

$$R = \left(\frac{1}{N} \right)^{1/3}, \quad (4)$$

where “ N ” is the concentration of total TMIs ($n_{\text{Fe}_2\text{O}_3} + n_{\text{V}_2\text{O}_5}$).

FIGURE 5: Effect of Fe_2O_3 content on activation energy W and pre-exponential factor $\log \sigma_0$.FIGURE 6: Variation of activation energy W with average ion separation R .

The small polaron radii “ r_p ” for the iron and vanadium ions have been calculated using [41]

$$r_p = \frac{1}{2} \left(\frac{\pi}{6N} \right)^{1/3}. \quad (5)$$

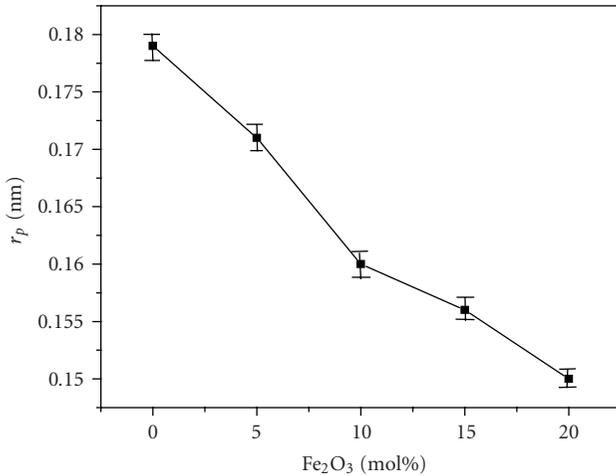


FIGURE 7: Variation of small polaron radius with iron content.

The calculated values of N , R , and r_p are tabulated in Table 4. The variation of activation energy W with average distance R , as shown in Figure 6, suggests that there is a prominent positive correlation between activation energy and average site separation. It is evident from Figure 7 that the small polaron (SP) radius decreases with the increase in Fe-ion concentration. From the above observations, it is concluded that on addition of Fe_2O_3 , conductivity increases, while the activation energy decreases due to the decrease in respective ion separation. These results are in good agreement with those reported by El-Desoky [36]. However, in the present investigation for sample $x = 10$ mol% of Fe_2O_3 , there are some minor changes observed in the density and electrical parameters as compared to our earlier investigation [42]. These changes may be attributed to difference in sample preparation conditions [11].

4. Conclusion

The structural and physical properties of $x\text{Fe}_2\text{O}_3$ -(40- x) B_2O_3 -60 V_2O_5 glass system, with $0 \leq x \leq 20$ (mol%), have been studied. No boroxol ring formation was observed in the structure of these glasses which suggest that glasses under study consist of randomly connected BO_3 and BO_4 groups. Addition of Fe_2O_3 produces NBOs in borate as well as in Vandate glass network along with the formation of VO_4 groups. The IR spectra of the present glass system indicate that Fe_2O_3 acts both, as a glass modifier ($x \leq 10$ mol%) as well as a glass former ($x = 15$ –20 mol%). The density of all the glass samples increases with Fe_2O_3 content as a result of the increase in NBOs due to the conversion of trigonal BO_3 structural units into BO_4 tetrahedral unit. In the temperature range 310–500 K, the variation of $\log \sigma$ with T^{-1} is approximately linear. It was observed that the dc conductivity increases with increasing iron content, and ranges from 5.435×10^{-4} to $4.194 \times 10^{-2} (\Omega\text{m})^{-1}$ at 400 K. The conduction in the present glass system was confirmed to be a result of primarily adiabatic hopping of small polarons between TMIs.

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