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High And Low Temperature DC Electrical Transport Properties of Vanadium Borate Glasses

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Abstract: **A series of Semiconducting borate glass samples doped with vanadium in the composition range $(H_3BO_3)_1 + (V_2O_5)_{1-x}$ where x ranging from 0.1, to 0.5 has been prepared by melt quench technique. The samples were annealed and their non-crystalline nature was confirmed by X-ray diffraction studies. Archimedes principle was adopted to measure room temperature density of the glass samples. High and low temperature DC electrical conductivity of the samples were analyzed and various polaron related parameters were investigated in the light of Mott's SPH for T > $\frac{\theta_D}{2}$ and Mott's VRH and Greaves VRH for T < $\frac{\theta_D}{2}$ (where θ_D Debye's temperature). The investigations in these glass systems showed to be non-adiabatic electrical conduction. As per as above theories, the calculated activation energies, conductivities and various polaron parameters were discussed. The detailed results of all the above parameters were discussed and it is for the first time the pure borate glasses doped with vanadium ions subjected to dc electrical conduction mechanism within the studied composition range.**

Keywords: **oxide glasses, single TM ion, high and low temperature, transport properties.**

1. INTRODUCTION

During the last few decades, researchers have shown considerable interest in glasses containing TM ions in single and mixed proportions. The presence of multivalent states of single transition metal ions in pure oxide glasses demonstrating semiconducting behavior and the conduction of electric current is due to polaron hopping from lower valance state of one transition metal ion site to higher valance state of another transition metal ion site between two same TM ions existing in glass matrix. The electron rich conduction TM ion doped oxide glass systems found substantial technological applications such as cathode materials for batteries, electrical switching devices, electronic circuit elements and gas sensors [1, 2, 3]. The electrical properties of single TM ion doped Vanado-phosphate glasses and containing with other glass formers also broadly studied and the ratio V^{4+} to V^{5+} institute the maximum electrical conductivity in these glass systems [4, 5]. The conduction mechanism in these glasses is due to hopping of charge carriers between two valence states of vanadium ion sites i.e. V^{4+} and V^{5+}. Different glass researchers have been reported the electrical properties of many single TM ion containing oxide glasses [6, 7, 8, 9,]. Many researchers also investigated the electrical conductivity mechanism in phosphate and borate glasses doped with TM ions in single and mixed proportion [9 to 11, 12]. However there are not many reports in the literature on electrical conductivity studies of pure borate glasses doped with single TM ions.

In the present research work we explore the room temperature density, molar volume, temperature independent high temperature dc activation energy and polaron related parameters under the shadow of Mott's SPH, and density of states at

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fermi level in the light of Mott's VRH and Greaves VRH in the following series of samples. Keeping in view of the fact that the following series of pure borate glasses doped with single TM ions such as vanadium are proposed for our research studies.

$(2H_3BO_3)_{1-x} + (V_2O_5)_x \longrightarrow B_2O_3 + V_2O_5 + 3H_2O$

x = 0.1, 0.2, 0.3, 0.4 and 0.5 labeled as RBV1, BV2, RBV3, RBV4 and RBV5 respectively.

2. BACKGROUND THEORY

The density of glass samples usually were determined by following the Archimedes principle,

$$D = \frac{w_{air}}{w_{air} - w_L} D_L \; g/cm^3 \quad (1)$$

The molar volume of the glass samples were determined using the following formula,

$$V_i = \Sigma \frac{x_i M_i}{D} \quad (2)$$

Where $V_i$molar volume is x_i is the mole fraction M_i is molecular weight D density of the particular glass sample.

The entire temperature dependent conductivity (either dc or ac temperature dependent but frequency independent conductivity) data measured from the experiment for all semiconducting oxide glass systems from known low temperature either room temperature or still at any lower temperature to any known high temperature to be divided into two regimes viz high temperature regime for $T > \theta_D/2$ where conductivity data exhibits liner dependence on temperature of the glass sample and low temperature regime for $T < \theta_D/2$ where the conductivity behaves nonlinear dependent on temperature of the sample. Where T is the temperature at which the high temperature conductivity data deviates from linearity and θ_D is known as Debye temperature [7, 13 to 23].

The VRH models which valid at very much lower temperatures i.e. $T < \theta_D/4$. However, Austin and Mott et al have been proposed that depending on the strength of the Coulombic interaction the density of states at the Fermi level changes with doping concentration of the composition and hence the VRH models may also applicable even at temperatures above 300k and higher temperatures [13, 21].

The semiconducting nature of electrical conductivity of TMI doped oxide glasses in the non-adiabatic regime can be theoretically investigated in the graceful of Mott's Small Polaron hopping (SPH) Model and fitted at high temperature regime i.e. for $T > \frac{\theta_D}{2}$ and Mott's and Greaves Variable Range Hopping (VRH) Models fitted for $T < \frac{\theta_D}{2}$ at low temperature regime [13 to 21].

2.1. Mott's Small Polaron (SPH) Model:

In our present investigation, the high temperature conductivity data i.e. when $T > \frac{\theta_D}{2}$ has been analyzed using SPH model. According to SPH [21, 22], the Non-adiabatic electrical conductivity of these glasses in the high temperature regime can be expressed as

$$\sigma = (\sigma_o/T)\, exp\,(-W/k_BT) \quad (3)$$

Where W is the activation energy and σ_o is the pre-exponential factor given as

$$\sigma_o = \nu_o N e^2 R^2 C(1-C) \exp[(-2\alpha R)/k_B] \quad (4)$$

Where $\nu_o = \theta_D k_B/h$ is optical phonon frequency [19], θ_D is Debye's temperature, *N* is concentration of TMI, *R* is Mean spacing between TM ion sites given by$R = N^{-1/3}$, $N = 2\,[(Dm_{TMI}/M_{TMI})N_A]$, D is density of the glass, m_{TMI} is mole fraction of TMI, M_{TMI} is the molecular weight of TMI, N_A is the Avogadro number, α is the tunneling factor and C is fraction of reduced TMI concentration to that of total TMI concentration [13, 24 to 28].

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2.2. Mott's Variable Range Hopping (MVRH) Model:

At low temperature regime the disorder energy perform a dominant role and Mott recommended that the hoping of electrons may occur outside the nearest neighbors. The expression for electrical conductivity as a consequence of VRH model is based on the single optical phonon approach [20, 21] and is given by

$$\sigma = A\, exp\left(-B/T^{-1/4}\right), \quad (5)$$

Where $A = 4\,[2\alpha^3/9\pi kN\,(E_F)]^{1/4}$, (6)

$$B = \left[e^2/2(8\pi)^{1/2}\right] \nu_o\, [N(E_F)/\alpha kT]^{1/2}. \quad (7)$$

2.3. Greaves Variable Range Hopping (GVRH) Model

The same low temperature regime can also be analysed in terms of Greaves VRH model [18]. Accordingly the expression for conductivity given as [19],

$$\sigma T^{1/2} = A\, \exp\left(-B/T^{-1/4}\right) \quad (8)$$

Where *A* and *B* are constants. *A* is given by (6) and

$$B = 2.1[\alpha^3/k_B N(E_F)]^{1/4} \quad (9)$$

3. EXPERIMENTAL TECHNIQUES

3.1. Synthesis of the glass samples:

The analytical grade chemicals boric acid (H_3BO_3) and vanadium oxide (V_2O_5) were procured from sigma Aldrich Company. The required amounts of weight's in mole fractions were weighed in citizen make single pan digital balance and thoroughly mixing by manually grinding in a agate then transferred in to silica crucible and heated up to their melting temperature in high temperature electrical muffle furnace up to maximum temperatures of 1200ºC-1300 ^{0}C. During the heating process the following chemical reactions can be expected to take place.

$(2H_3BO_3)_{1-x} + (V_2O_5)_x \longrightarrow B_2O_3 + V_2O_5 + 3H_2O$

x = 0.1, 0.2, 0.3, 0.4 and 0.5 labeled as RBV1, RBV2, RBV3, RBV4 and RBV5 respectively.

The gasses and vapour, which were the byproducts of the above chemical reaction, got liberated around 500°C. The mixture was heated at this temperature was increased to just above 1300°C where the mixture got melted and remained there for more than half an hour. When the melt was looking like a transparent liquid it was quickly quenched to room temperature by pouring it on to a stainless steel plate and covering with another stainless steel plate. The random sized pieces of glasses thus formed were collected.

3.2. Annealing:

In the annealing process the samples were heated at 350°C in a closed muffle furnace for an hour and allowed them to cool over 24 hours. The annealed samples were used for experimental measurements.

3.3. XR diffraction studies:

The present glasses were subjected to X-ray diffraction studies at Indian Institute of Science, Bangalore. XRD patterns of the all the samples were taken by using Cu-Ku radiation of wavelength 1.5418 Å in Phillips PW3710 diffractometer.

3.4. DC conductivity measurement:

Annealed glass samples after obtaining required dimensions were selected for dc conductivity studies. The electrodes for these measurements were made by applying silver conducting paint on top and bottom surfaces of the samples. The resistance was measured by following two-probe method. A constant dc voltage was applied across the sample and current passing through the sample was measured with the help of a Keithley make DMM. The voltage was measured

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using a voltmeter to the accuracy of ±10mV. Temperature was measured using a chromel-alumel thermo-couple and a micro voltmeter to the accuracy of ±1°C. These measurements were performed as a function of temperature. Dimensions of the samples were determined using a traveling microscope with a least count of 0.001cm. These conductivity measurements were carried out for the temperature range 323K to 653K.

Using the measured electrical resistance, R, and dimensions of the sample the conductivities, σ were determined as per, $\sigma = (1/\rho)$, where resistivity, $\rho = (RA/t)$, A is cross sectional area and t is thickness of the sample. The error on conductivity, $\Delta\sigma$, was estimated taking into account of errors on the measured voltages, ΔV, and currents, ΔI, according to the relation, $\Delta\sigma = (\Delta\rho/\sigma^2)$, where error on resistivity, $\Delta\sigma = \Delta r(A/I)$, $(\Delta r/r) = (\Delta V/V) - (\Delta I/I)$. The estimated errors on conductivity are found to lie in the range of 3% to 4%.

4. RESULTS AND DISCUSSION

4.1. X-ray Diffraction:

All prepared samples subjected to X-ray diffraction studies and In order to save space only the XRD pattern of two samples i.e. from first series vanadium doped borate glasses RBV1 and RBV5 two samples. And all glass samples of series which are also exhibited same nature. Based on the XRD pattern shown in Fig.1, it is taken for granted that the present glasses are non-crystalline in nature [29].

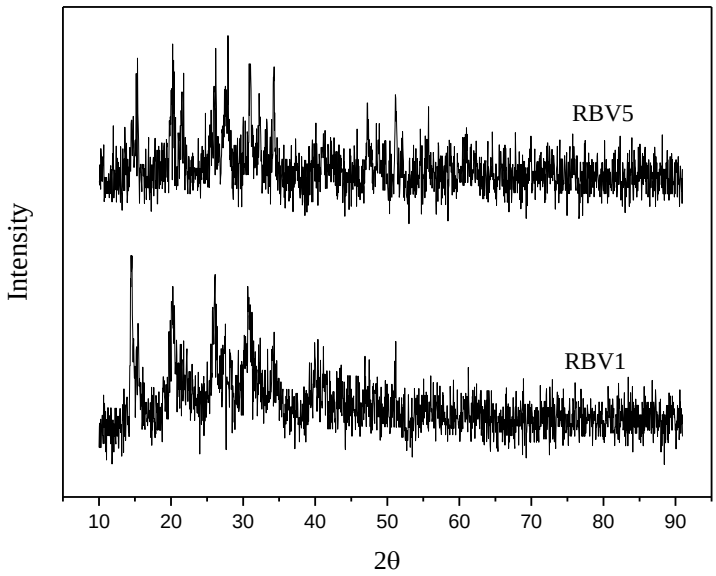

Fig 1: XRD Pattern of RBV1 & RBV5 glass samples of RBV glass system.

4.2. Density discussion:

The room temperature density of annealed all glass samples were determined using Archimedes principle and the measured density of vanadium doped borate glasses lies between the values 1.2311 gm/cm^{-3} to 3.880 gm/cm^{-3} and recorded in table 5.1. The density of vanadium doped borate glasses increases with increase of transition metal ion content and the calculated values of density values similar to many TM ion doped oxide glasses [8, 30 to 32]. The molar volume of these glasses are ranging from 33.304 to 124.095 and decrease with increase of vanadium (TMI) concentration which reveals that the variation of density and molar volume are similar behaviour as that of many TM ion doped oxide glasses. The average distance "R" between the transition metal ions (homogenous TMI distribution in the glass matrix the volume is assumed) was calculated as follows,

$$R = \left(\frac{1}{N}\right)^3 \quad (10)$$

Where "N" is the concentration of total TM ion which could be calculated by using the relation $N = 2\,[(Dx_i/M_i)*N_A$ where D is the density of the glass samples, x_i is mole fraction of TMI, M_i molecular weight of respective ions, N_A is the Avogadro's number.

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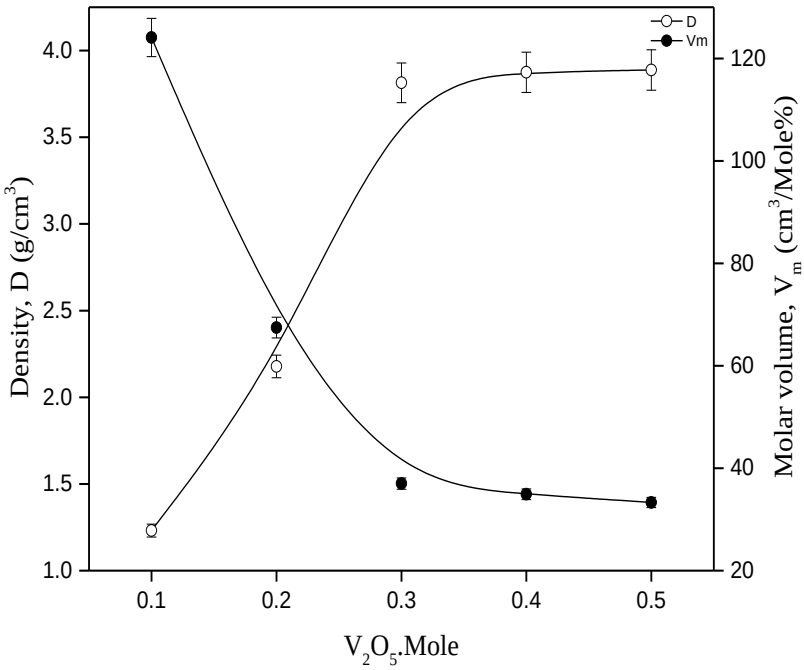

Fig 2: Compositional variation of density, r and Molar volume, V_m of RBV glasses.

The variation in density and molar volume with increase of vanadium concentration (TMI) of the RBV series of glasses reveals that the glass network continuously close or tight packed in the studied composition range. The monotonous decrease in molar volume V_M with TM ion concentration indicates that there is no substantial change taking place in the topology of glass network in the studied series [30, 33].

Table 1: physical properties of RBV series glasses

B_2O_3 1-x	V_2O_5 x	Glass	D (gm/cm^3)	V_m (cm^3/mol)	$N \times 10^{21}$ (cm^{-3})	R (nm)	θ_D (K)
0.9	0.1	RBV1	1.231	124.095	0.785	1.084	716
0.8	0.2	RBV2	2.178	67.468	2.778	0.711	786
0.7	0.3	RBV3	3.814	37.000	7.297	0.516	816
0.6	0.4	RBV4	3.875	34.918	9.884	0.466	836
0.5	0.5	RBV5	3.888	33.304	12.400	0.432	886

4.3. DC conductivity:

4.3.1. The dc conductivity discussion of glass samples:

According to Mott's Small Polaron hopping model the electrical conductivity in the non-adiabatic conduction as per as the equation 3.4 the plots of ln (σT) verses (1/T) were plotted for the present series of glass samples and depicted in Figs.5.5. From these figures it is observed that curves are reasonably linear in the high temperature region and nonlinear in the low temperature region. Therefore, the least square linear lines were fit to the data in the high temperature region that is above $T > \theta_D/2$, using origin graphics- version 7.1. The degree of linear fits was noted by noting correlation coefficient, Y, from the graphs and it was 0.9998 to 0.9995 for all the glasses. The high temperature activation energy for all the samples of RBV series above the temperature regime $T > \theta_D/2$ at which the curve or the data deviates from the linear line are determined from the figures. The quantity T is related to Debye's temperature, $\theta_D = 2T$. The values of θ_D for all the glasses have been estimated.

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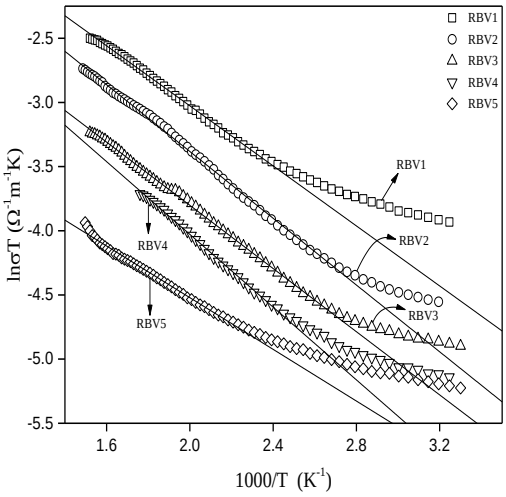

Fig 3: Plots of ln (σT) vs 1000/T of RBV glasses. Solid lines are the least square fits to the high temperature data.

The E_{dc} values were determined from the slopes of these lines and they were found to be in the range of 0.085eV to 0.123eV. The conductivity values at temperature 513k measured was in the range $2.213x10^{-5}$(ohm-m)$^{-1}$ to $1.001x10^{-4}$ (ohm-m)$^{-1}$. Similar low values were reported in literature and these sizes of high temperature activation energy E_{dc} of present series of glass systems are smaller than other TMI doped oxide glass systems reported in literature [34 to 39]. The smaller values of activation energy and higher magnitudes of conductivity in these glasses might be considered as the effect of partial charge of the cations of the glass forming oxides (Bi_2O_3, Cao, B_2O_3, V_2O_5) on activation energy for hopping conduction in the TMO doped glasses [40]. The high activation energy decreases and conductivity increases with increase of mole fractions of vanadium which is similar to single TMI doped oxide glasses reported [34 to 39] and plotted in the fig. 4.

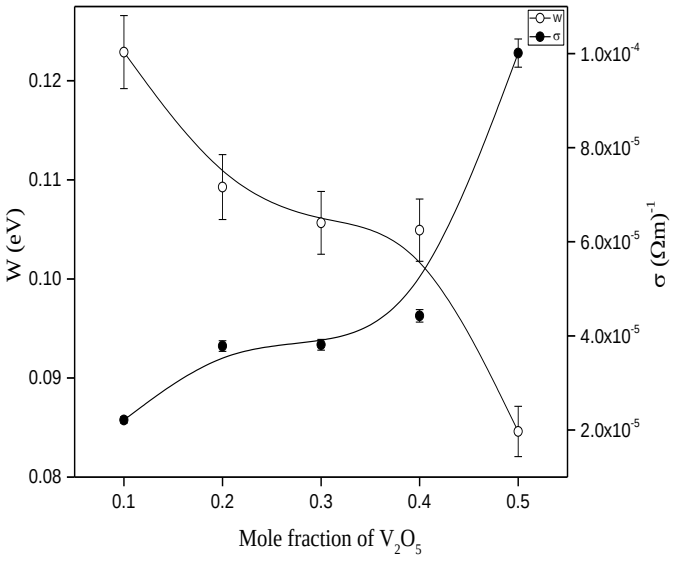

Fig 4: Variation W_{DC}, conductivity, σ, at 513K with mole fraction V_2O_5 of RBV glasses. (Solid lines are guides to the eye).

4.3.2. Various polaron related parameters discussion:

Considering the strong electron phonon interaction the activation energy in the high temperature region where nearest neighbor thermally activated hopping predominates the polaron hopping energy W_H and disorder energy W_D which might be exists between initial and final sites of ions in the glass matrix due to variation in the local environment of the ions. Austin and Mott [13] proposed,

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$W = W_H + W_D/2 \qquad T > \theta_D/2$ (11)

$\approx W_D \qquad T < \theta_D/4$ (12)

Where W_H is the polaron hopping energy and W_D is the disorder energy. The polaron hopping energy W_H was calculated using the formula [37],

$$W_H = W_P/2 = (e^2/4\varepsilon_p)(r_p^{-1} - R^{-1}) \quad (13)$$

Where W_P is the polaron binding energy, ε_p is the effective dielectric constant and which can be determined using the equation [22],

$$\varepsilon_p = e^2/4Wr_p \quad (14)$$

Where r_p is the small polaron radius which can be calculated from the following relation [38],

$$r_p = (1/2)(\pi/6N)^{1/3} \quad (15)$$

The estimated values of the above physical parameters are listed in table 2 and they are comparable with reported for other similar TMI doped oxide glasses [33, 40]. The non-adiabatic electrical conduction in the present series of glasses could be understood by applying Holestien condition, because the pre exponential factor is various with doping concentration of the TMI and from which for small polaron hopping model the polaron bandwidth is given by the relation [37],

$$J \begin{cases} > (2kTW_H/\pi)^{1/4}(h\nu_0/\pi)^{1/2} & \text{for adiabatic SPH conduction} \\ > (2kTW_H/\pi)^{1/4}(h\nu_0/\pi)^{1/2} & \text{for non-adiabatic SPH conduction} \end{cases} \quad (16)$$

The polaron band widths were calculated from the relation $j=j_0exp(-\alpha R)$, where $j = W_{H(min)}/4$ [38] and recorded in table and the of the value $\alpha=20\ nm^{-1}$ was taken from the reported literature for many TMI doped glasses [19]. From table 2 and 3 it can be noted the value of J satisfies the non-adiabatic SPH conduction for all the present series of glasses given in equation (5.8) and Holestein condition i.e. $J < W_H/3$ [20] for formation of small polaron. The density of states N (E_F) at Fermi level were determined by the following relation [37],

$$J = e^3N(E_F)^{1/2}/(\varepsilon_P)^{3/2} \quad (17)$$

And recorded in table 5.4. The calculated values which are recorded in table 5.4 and of the order of 10^{27}/ev-m^3 to 10^{28}/ev-m^3 and were good agreement with reported values for many TMI doped oxide glasses [8, 34 to 38].

Table 2: various polaron related parameters

Glass	W (eV)	σ (Siemen)	r_p (nm)	ε_p	W_H (eV)	W_P (eV)	W_D (eV)
RBV1	0.123	2.21310^{-5}	0.4369	84.247	0.0733	0.1467	0.0990
RBV2	0.109	3.78410^{-5}	0.2867	144.382	0.0652	0.1304	0.0880
RBV3	0.106	3.81210^{-5}	0.2078	206.034	0.0630	0.1261	0.0851
RBV4	0.105	4.42610^{-5}	0.1878	229.561	0.0626	0.1252	0.0845
RBV5	0.085	1.00110^{-4}	0.1742	307.043	0.0505	0.1010	0.0681

The small polaron coupling constant γ_P which is measure of the electron phonon interaction and for the present series of the glasses could be calculated by the following relation, $\gamma_P = \frac{W_P}{\hbar\omega_0}$ where W_p is the polaron binding energy and is approximately given by $W_p = 2W_H$ and $\hbar = \frac{h}{2\pi}$, $\omega_0 = 2\pi\nu_0$ then using the values of W_H and ν_0 the values of γ_P were determined for the series of glass samples which single TM ion doped glass systems and recorded in table 3. Austin and Mott [13] suggested that the value of $\gamma_P > 4$ indicates strong electron phonon interaction in the solids. In these glass system the value of electron phonon coupling constant go on decreasing from 4.7574 to 2.6464 and conductivity of the glass samples increasing with increase in doping concentration of vanadium ions and the values were similar for many TMI doped glasses reported in literature [34 to 38, 41].

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The temperature independence of thermopower (at room temperature and above) denotes that all available carriers are mobile and in these glass systems take V^{4+} concentration [42, 43] in such a case the mobilities of carriers were calculated for these glasses by using the relation $\sigma = ne\mu$ where n is the concentration of the charge carries, μ is the mobility of charge carrier in this case polaron and e is the electron charge. The mobility values are very low and ranging 6.3717E-05 m^2/vs to 9.7110E-06 m^2/vs and in agreement with previous results obtained for many semiconducting oxide glasses [42-44]. These low values of mobilities are due to localization processes which occur usually in such materials (i) a strong interaction of the unpaired electron with the polar network leading to the formation of small polaron [7] and (ii) a localization of the carriers which arises from the structural disorder and corresponds to the well-known "Anderson localization" [45]. An examination of the semi-conduction mechanism in the above system of glasses suggest that generally a small polaron hopping model is adequate.

Table 3: various polaron related parameters

Glass	[a]J (eV)	[b]J (eV)	[c]J (eV)	v_0 (Hz)	γ_p	$N(E_F)$ ($eV^{-1}m^{-3}$)	μ at 553K (m^2/vs)
RBV1	0.012	0.0166	0.0168	7.4561×10^{12}	4.757	$2.833x10^{28}$	$6.371x10^{-5}$
RBV2	0.018	0.0207	0.0245	8.1850×10^{12}	3.853	$5.013x10^{28}$	$1.864\ x10^{-5}$
RBV3	0.016	0.0207	0.0217	8.4974×10^{12}	3.588	$3.938\ x10^{28}$	$1.205\ x10^{-5}$
RBV4	0.015	0.0209	0.0210	8.7057×10^{12}	3.478	$3.969x10^{28}$	$1.055\ x10^{-5}$
RBV5	0.015	0.0208	0.0209	9.2264×10^{12}	2.646	$9.591x10^{28}$	$9.711\ x10^{-5}$

[a]J-calculated from formula, [b]J- condition for non-adiabatic SPH electrical conduction, [c]J- Holestein condition for Small Polaron Formation.

4.4. Low temperature dc conductivity of present series glass samples:

According to Mott's variable range hopping model as per as equation 5 the plots of ln (σ) vs $T^{-1/4}$ and Mott and Greaves variable range hopping model as per as equation 8 the plots of ln ($\sigma T^{1/2}$) vs $T^{1/4}$ to the remaining data below $\theta_D/2$ for first series RBV samples where the data is linear were plotted and as depicted in figures 6 and 7. From the *figure 5* the values of the constants A and B were determined from the slopes of the ln (σ) vs $T^{-1/4}$ and $N(E_F)$ values have been calculated from equations 7 and 9 assuming $\alpha=20(nm)^{-1}$ [19] for TMI glasses. The data can be well described by the least square linear lines. The values of $N(E_F)$ are the order of 10^{32} which are very larger values than for many TMI glasses [46, 47].

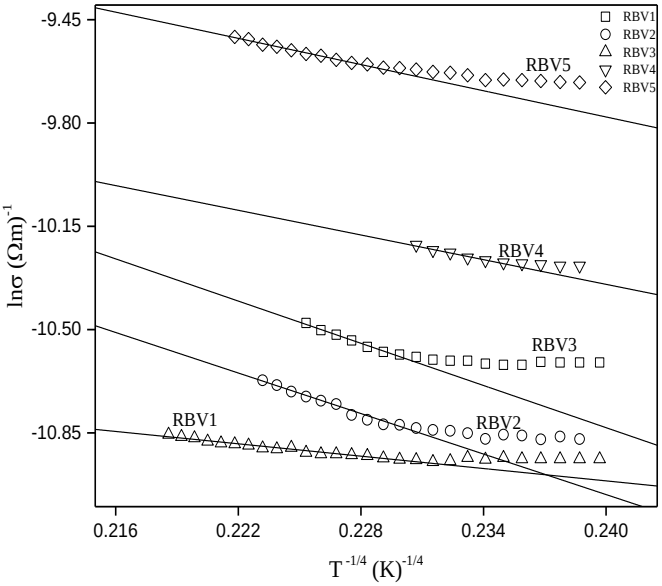

Fig 5: Plots of ln (σ) Vs $T^{-1/4}$ RBV glasses. Solid lines are the least square linear fits to the low temperature data.

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According to Greaves VRH model which the graph were plotted in figure 6 from which the values of A and B were calculated. From the values B the density of states were calculated for all the samples in the first series of glass samples as recorded in table 4 and the values of $N(E_F)$ are of the order of 10^{30} which are almost comparable with the reported values in the literature for many TMI glasses [46, 47]. The $N(E_F)$ values obtained from Greaves VRH model are in close agreement with the literature and it may be proposed that that at temperature below $\theta_D/2$ Greaves VRH model valid for the present series of glasses.

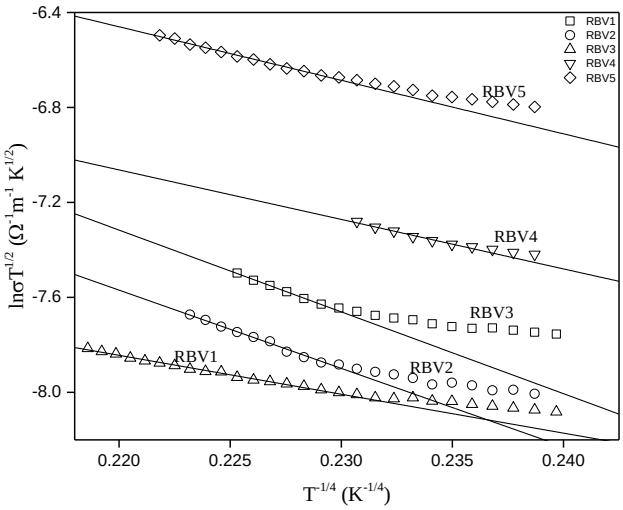

Fig 6: Plots of ln sT1/2 Vs T1/4 of RBV glasses. Solid lines are the least square fits to the data

Table 4: Mott and Greave's parameters for VRH conduction in RBV glasses

Glass	$N(E_F)$ Density of states due to MVRH ($eV^{-1}m^{-3}$)	$N(E_F)$ Density of states due to GVRH ($eV^{-1}m^{-3}$)
RBV1	2.2122×10^{32}	1.351×10^{30}
RBV2	8.3637×10^{32}	1.354×10^{30}
RBV3	1.2270×10^{33}	6.466×10^{30}
RBV4	2.3775×10^{33}	7.389×10^{30}
RBV5	3.4374×10^{33}	2.645×10^{31}

5. CONCLUSIONS

A series of five samples of borate glass doped with single TM ion i.e. vanadium were prepared by conventional melt quench technique. The samples were annealed at a temperature 300ºC and the non-crystalline nature of the glass samples were confirmed by XR diffraction studies. The room temperature density was measured by Archimedes principle and molar volume estimated. The density increases and molar volume decreases with increase of vanadium ion concentration, which reveals that no topology of the glass samples changes with addition of vanadium ion concentration into the glass matrix. The measured dc conductivity increases and high temperature activation energy decreases with increase of vanadium ion concentration. Various polaron parameters were estimated for present series of glass samples and discussed in detail. The results leads in understanding that the transport properties in high temperature regime that is the validity of Mott's SPH model, small polaron formation, non-adiabatic electrical conduction, multi phonon assisted motion of polarons in these materials, electron phonon coupling constant, mobility of electrons etc. The low temperature data were verified in the light of MVRH and GVRH and results indicates that GVRH was adequate to explain the low temperature data in the present series of glass samples.

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