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Transport properties of semiconducting Fe₂O₃–PbO–B₂O₃ glasses*

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Abstract. Density, DC conductivity and ¹¹B wide-line NMR investigations have been carried out for $xFe_2O_3-(50 - x)PbO-50B_2O_3$ glasses ($0 \le x \le 20$ mol%). The NMR spectra indicated the formation of non-bridging oxygen ions in the borate network upon introducing Fe₂O₃. The DC conductivity and the activation energy for conduction change markedly with Fe₂O₃ content. The high-temperature conductivities can be described by Mott's model for the phonon-assisted hopping of small polarons. The polaron hopping is shown to be non-adiabatic. The lowtemperature conductivities are characterized by the variable-range hopping mechanism. For Fe₂O₃ contents greater than 15 mol%, glass-crystal composites are formed, changing greatly the electrical properties, and other conduction mechanism is to be assumed.

1. Introduction

The structure [1, 2] and the electrical properties [3-5] of oxide glasses containing transitionmetal ions, especially those containing iron ions [6-9] have been studied with increasing interest in the last few years. The general condition for semiconducting behaviour of such glasses is the presence of the transition-metal ion in more than one valence state. It is believed [10-13] that the electrical conduction in these glasses is due to a strong electronphonon interaction which results in the formation of small polarons. The conductivity and the transport properties in general are controlled with the hopping of small polarons or electrons between localized states [3, 11, 14] (the low- and high-valence sites).

Previously we investigated the structure and the electrical conduction in $Na_2O-V_2O_5-B_2O_3$ glasses [15] and PbO- $V_2O_5-B_2O_3$ glasses [16]. Increasing V_2O_5 in these glasses causes a decrease in the activation energy and an increase in the conductivity. However, the latter was found to depend mainly on the oxygen-to-boron ratio. In the present work it is aimed to investigate the structure and the DC conductivity of some Fe₂O₃-PbO-B₂O₃ glasses, with special attention to applicability of Mott's model for electrical conduction in these glasses.

2. Experimental details

 xFe_2O_3 -(50 - x)PbO-50B₂O₃ glasses (table 1) were prepared using reagent-grade Fe₂O₃, PbO and H₃BO₃. The glasses were melted in porcelain crucibles in an electric furnace at temperatures between 900 and 1050 °C, depending on the glass composition. To assure

* Dedicated to Professor Dr W Müller-Warmuth (Münster, Germany) on the occasion of his 65th birthday.

_	Comp	position (n	nol%)	D	V		117
Glass	Fe ₂ O ₃	PbO	B ₂ O ₃	(g cm ⁻³)	$(\text{cm}^3 \text{ mol}^{-1})$	N ₄	(eV)
G1	0.0	50	50	5.199	28.16	0.50	1.32
G2	1.25	48.75	50	5.190	27.97		1.02
G3	2.5	47.5	50	5.180	27.79	0.39	0.92
G4	5.6ª	45.0	50	5.145	27.96	0,31	0.78
G5	7.5ª	42.5	50	5.073	27.82		0.73
G6	10.5ª	40.0	50	4.991	28.10	0.23	0.70
G7	12.7ª	37.5	50	4.844	28.50		0.72
G8	15.9ª	35.0	50	4.527	30,29	0.21	0.73
G9	17.5	32.5	50	_	—		0.26
G10	20.0	30.0	50		<u> </u>		0.22

Table 1. Compositions, densities D, molar volumes $V_{\rm m}$, the fractions N_4 of four-coordinated boron ions and the high-temperature activation energies W for the glasses studied.

^a Chemically analysed concentrations.

homogeneity the well mixed components were added in small portions; the melt was swirled frequently and then maintained until considered homogeneous. The homogenized melt was poured onto a steel plate and the glass was transferred to an annealing furnace for subsequent cooling at a rate of about 40 °C h^{-1} .

X-ray diffraction patterns indicated that there is a crystalline structure in the glasses containing more than 15 mol% Fe_2O_3 , whereas the glasses with lower Fe_2O_3 contents appear to be amorphous.

Plates of about 0.2–0.3 cm thickness were used to measure the DC conductivity. A graphite coating was applied to the flat surfaces to serve as electrodes. The coated area has a 0.5 cm radius. The resistance was measured using an insulation tester type TM14 (Level Electronics Ltd) with a $10^3-10^{13} \Omega$ range. The applied potential difference ranged between 3 and 15 V. As a rule, three samples of each glass were used to measure the resistance. The experimental error in determining the activation energy for conduction is estimated to be less than 0.02 eV, whereas the relative error in the conductivities is expected to be $\pm 5\%$.

Atomic absorption spectrometry (Perkin–Elmer 2380 spectrometer) was used to determine the total iron content in the glasses. The concentration of Fe^{3+} was determined by means of polarography (Polarecord E 506, Metrohn Herisan, connected with a polarography stand E 505). A suitable supporting electrolyte [17] was used.

A Varian (4210 A) wide-line NMR spectrometer was used to obtain the ¹¹B NMR spectra. A magnetic field strength of 7 T was used. The NMR signals were measured as derivative curves of the absorption lines and averaged. A computer simulation of the total ¹¹B NMR response was used to obtain the fraction N_4 of four-coordinated and the fraction N_3 of three-coordinated boron ions. The N_4 -values obtained are given in table 1.

The densities were determined at room temperature using the Archimedes method with xylene as the immersing liquid. Three samples of each glass were used to determine the density. The density values (table 1) were reproducible to ± 0.01 g cm⁻³.

3. Results and discussion

3.1. Structural aspects

The temperature dependence of conductivity for the glasses investigated is given in figure 1. Except for G1-G3, there are two regions for the conductivity change. In the first the

conductivity decreases linearly with increasing reciprocal of the absolute temperature, indicating an activated behaviour in the high-temperature region. In the low-temperature region the change is non-linear which exhibits a temperature-dependent activation energy. This behaviour represents a characteristic feature of the small-polaron hopping conduction [10, 11, 18, 19]. For G4-G8 the activation energy decreases with decrease in temperature. The opposite behaviour is observed for the glasses G9 and G10.





Figure 1. Temperature dependence of the conductivity for the glasses investigated.

Figure 2. Variation in the activation energy W and $\ln \rho_{473 \text{ K}}$ with the Fe₂O₃ content for glasses G1-G10.

In figure 2 is shown the dependence on Fe₂O₃ of both the high-temperature activation energy W (T > 480 K) and the natural logarithm of resistivity (at 473 K). The activation energy decreases rapidly upon introducing Fe₂O₃ into the structure (up to 5 mol%) and it seems to have a constant value for 7.5 mol% \leq Fe₂O₃ \leq 15 mol%. A sudden decrease in W is observed for a Fe₂O₃ content greater than 15 mol%. The resistivity ρ behaves similarly to W as the Fe₂O₃ concentration changes. These sudden changes in W and ln ρ may reflect structural changes in these glasses.

In Fe_2O_3 -PbO-B₂O₃ glasses, both Fe_2O_3 [20] and PbO [21,22] enter the structure in two forms: as a network former and/or a network modifier. The relative concentration of the network-forming oxide depends on the glass composition. In PbO-B₂O₃ glasses, lead enters

(at low concentrations) as modifier Pb^{2+} ions (PbO_m) at the rate of two BO₄ tetrahedra for each Pb^{2+} ion [21]. PbO_4 pyramids (network-forming lead oxide, PbO_f) which start to form above 15–20 mol% PbO are assumed to bridge preferentially to BO₃ rather than to BO₄ units. For 20 mol% < PbO ≤ 50 mol%, the fraction of PbO_m decreases with increase in PbO content [16]. Similarly, the fraction of network-modifying FeO in Fe₂O₃–PbO–B₂O₃ glasses [23] decreases as Fe₂O₃ is increased.

Figure 3 shows the fraction N_4 of four-coordinated boron ions as a function of the Fe₂O₃ content. The decrease in N_4 as the oxygen-to-boron ratio increases reveals that non-bridging oxygen ions (NBOs) are formed in the borate network with increasing Fe₂O₃.



Figure 3. The fraction N_4 of four-coordinated boron ions as a function of the Fe₂O₃ content for xFe_2O_3 - $(50 - x)PbO-50B_2O_3$ glasses.

Figure 4. Change in $\ln \sigma_0$ with the average Fe-Fe distance R for the glasses G4-G8.

The N_4 -values can be used (for glasses containing low Fe₂O₃ contents) to calculate the mole fraction of modifying oxide (MO) in glass, where MO = FeO+PbO_m. Raman spectroscopy investigations on PbO-B₂O₃ glasses [24] indicated that, above 50 mol% PbO, four-coordinated boron ions convert into three-coordinated boron ions. The latter exist mainly in chain- and ring-type metaborate units (BO₃ groups with one NBO). This process takes place up to 70 mol% PbO. MO can be obtained considering that each FeO or PbO_m molecule may convert two BO₄ tetrahedra into two asymmetric BO₃ groups (NBOs) and that two Fe²⁺ ions can be produced from a modifying Fe₂O₃ molecule. The MO can be calculated using the relation

$$MO = (1 - N_4)/2.$$
(1)

The determined N_4 -values and the analysed FeO concentrations (table 1) are used to calculate the molar concentration of PbO_m. The PbO_m-values obtained are relatively higher (about 10% in G5–G7) than those in the comparable PbO–B₂O₃ glasses [16]. This result may indicate that, upon introducing Fe₂O₃ into the glasses studied, PbO_f gives way to PbO_m which contributes with FeO to the formation of NBOs. A similar conclusion was obtained for ZnO–PbO–B₂O₃ glasses [25], where the increase in ZnO at the expense of PbO leads to a decrease in the fraction of PbO_f in glass.

3.2. DC conductivity

The electric conduction in G1, which is free of Fe₂O₃, is proposed to be of ionic nature and Pb²⁺ ions are assumed to be the charge carriers [16]. In such glasses the conductivity σ is given [26] as

$$\sigma = (nq^2 s d^2 f / 6k_{\rm B}T) \exp(-W/k_{\rm B}T)$$
⁽²⁾

where *n* is the concentration of the mobile ions, *q* is the ionic charge, *s* is the number of interstices (holes) directly surrounding the mobile ion, *d* is the average distance between the holes, *f* is the vibration frequency of the ion, *W* is the activation energy for conduction, $k_{\rm B}$ is the Boltzmann constant and *T* is the absolute temperature.

The dependence of $\ln \sigma$ on 1/T is linear for the glasses G1-G3. It seems that the concentration of Fe₂O₃ in G2 and G3 is insufficient to show a deviation from linearity as in the case of G4-G10 (figure 1). However, W decreases markedly when introducing Fe₂O₃ into the structure.

The conduction processes in glasses containing transition-metal ions have been studied by Mott [10] in terms of phonon-assisted hopping of small polarons between localized states. According to the model of Mott and co-workers [10–13] the DC conductivity σ for an oxide semiconductor in the high-temperature region ($T > \Theta_D/2$, where Θ_D is the Debye temperature) is characterized by nearest-neighbour hopping and is given by

$$\sigma = (v_0 N e^2 R^2 / k_B T) a (1 - a) \exp(-2\alpha R) \exp(-W / k_B T)$$
(3)

where ν_0 is the longitudinal optical phonon frequency, N is the number of transition-metal ion sites per unit volume, e is the electronic charge, R is the average site separation, a is the ratio of the concentration of transition-metal ions in the low-valence state to the total concentration of the transition-metal ions, i.e. the fraction of sites occupied by an electron and α is the decay parameter for the wavefunction describing the localized state at each site. The term $\exp(-2\alpha R)$ represents the electron overlap integral between the localized states or the probability of tunnelling.

The average site separation R (table 2) can be calculated using the density data of the glasses studied and the analysed total iron ion concentration (table 1), where $R = N^{-1/3}$. The rate α of wavefunction decay can be determined when plotting $\ln \sigma_0$ against R (figure 4) where

$$\sigma_{\rm o} = (\nu_{\rm o} N e^2 R^2 / k_{\rm B} T) a (1-a) \exp(-2\alpha R)$$
(3a)

Oľ

$$\sigma_{\rm o} = (\nu_{\rm o} e^2 / k_{\rm B} T R) a (1-a) \exp(-2\alpha R). \tag{3b}$$

This relation can be rewritten as

$$\sigma_{\rm o} = E \exp(-2\alpha R) \tag{3c}$$

with

$$E = (v_0 e^2 / k_{\rm B} T R) a (1-a).$$
(3d)

Figure 4 shows a linear dependence of $\ln \sigma_0$ on R, giving $\ln E = 5.32$. For G4-G8, the factor a(1-a) has nearly a constant value (between 0.23 and 0.25). Therefore, the quantity

Glass	Analysed total iron ion concentration N $(10^{21} \text{ cm}^{-3})$	Analysed Fe ²⁺ concentration (10 ²¹ cm ⁻³)	Reduced iron ion ratio a	Average Fe–Fe distance <i>R</i> (Å)
G4	2.41	0.91	0.38	7.46
GS	3.25	1.38	0.43	6.75
G6	4.50	1.63	0.36	6.05
G7	5.37	1.90	0.35	5.71
G8	6.32	3.02	0.48	5.41

Table 2. Some parameters of the glasses G4-G8.

 $v_0 e^2/k_B T R$ should represent a constant. This means that an increase in *T*, and subsequently in *R*, should be compensated by an increase in v_0 . Consequently, *E* can be considered as a temperature- and *R*-independent constant for the glasses G4-G8. From the slope of the straight line (figure 4), α was found to be 0.73 Å⁻¹. This value reflects strong localized states [12] in the glasses investigated.

The longitudinal optical phonon frequency v_0 can be estimated from equation (3), by using the high-temperature conductivity data. The phonon frequencies are of the order of 10^{13} s⁻¹, very close to the frequencies of the lattice vibrations [10, 27]. It must be noted that, in calculating the v_0 -values, the effect of temperature on *R* was not taken into consideration. From equation (3), one obtains v_0 as

$$v_0 = [k_{\rm B}/e^2 a (1-a)] [R/\exp(-2\alpha R)] [\sigma T/\exp(-W/k_{\rm B}T)].$$
(3e)

The first term on the right-hand side in this equation has a constant value. Owing to an expected increase in R with increasing T, both the second and the third terms increase with increase in temperature. The second term is more sensitive to changes in R compared with the change in the third term with T. Using R = 7.46 Å (the room-temperature value) for G4, v_0 was found to increase from 1.27×10^{13} s⁻¹ at 493 K to 2.47×10^{13} s⁻¹ at 613 K.

The polaron binding energy W_p is given [11] as

$$W_{\rm p} = e^2 / 2\epsilon_{\rm p} r_{\rm p} \tag{4}$$

where ϵ_p is the effective dielectric constant and r_p is the polaron radius. The latter can be estimated using the formula derived by Bogomolov *et al* [28]. They indicated that for a non-dispersive system the polaron radius is given by

$$r_{\rm p} = (\pi/6N)^{1/3}/2. \tag{5}$$

Using $R = N^{-1/3}$, equation (5) can be reformulated as

$$r_{\rm p} = 0.403R.$$
 (5a)

Equation (5), which was derived for crystalline TiO₂, is found to be applicable in the case of complex glass systems [5,9]. Considering that the polaron binding energy W_p can be taken as twice the high-temperature activation energy W [9], the ϵ_p -values can be calculated from equation (4) when using the estimated r_p -values (table 3). The r_p - and ϵ_p -values obtained are comparable with those found for other glass systems [5,9].

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Tab	Нή	

	Ø523 K	σ_0	'n,		Vo(523 K)	W_{H}	$N(E_{\Gamma})^{a}$
Glass	$(10^{-9} \ \Omega^{-1} \ \text{cm}^{-1})$	$(10^{-3} \Omega^{-1} cm^{-1})$	(¥)	¢	(10 ¹³ s ⁻¹)	(eV)	$(10^{19} \text{ eV}^{-1} \text{ cm}^{-3})$
5	0.11	3.70	3.01	1.54	1.8	0.46	0.0
S	1.08	12.53	2.72	1.82	1.7	0.43	1.1
S	5.33	28.72	2.44	2.11	1.5	0.42	1.5
6	6.13	44.60	2.30	2.18	1.6	0.44	1.3
89	6.98	76.54	2.18	2.27	1.2	0.43	1.1
^a Value	s for low-slone regions	t in figure 5.					
	•	'n					

Values for low-slope regions in fig
 Values for higher-slope regions.

0.1 0.8 3.0 2.7

0.5 0.6 0.8 0.7

 $\mu_{\rm H(523 \ K)}$ (10⁻¹¹ cm² V⁻¹ s⁻¹)

 $N(E_{\rm F})^{\rm b}$ (10¹⁹ eV⁻¹ cm⁻³)

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The polaron hopping energy $W_{\rm H}$ and the disorder energy $W_{\rm D}$ (which arises from the energy difference of the neighbouring sites) are related to the activation energy W by the relation [11]

$$W = \begin{cases} W_{\rm H} + W_{\rm D}/2 \\ W_{\rm D} \end{cases} \quad \text{for } \begin{cases} T > \Theta_{\rm D}/2 \\ T < \Theta_{\rm D}/4. \end{cases}$$
(6)

The hopping energy $W_{\rm H}$ can be calculated using the relation [11]

$$W_{\rm H} = (e^2/4\epsilon_{\rm p})(1/r_{\rm p} - 1/R). \tag{7}$$

Using the estimated values of $W_{\rm H}$ (table 3), one obtains values for $W_{\rm D}$ of around 0.6 eV for the glasses G4-G8. This value agrees with those obtained for different Fe₂O₃-containing glasses [9]. However, such a value is much higher than the values calculated theoretically which are in the range of 0.1 eV [9]. The difference was attributed to the presence of two possible coordinations (4 and 6) for the iron ions in such glasses.

Mott [13] indicated that the linear dependence of $\ln \sigma$ on 1/T is expected only when hopping of the charge carriers is between nearest neighbours. At low temperatures, tunnelling to more distant sites may become favourable and this causes what is known as the variable-range hopping mechanism. The conductivity-temperature dependence in the low-temperature region can be represented by

$$\sigma = A \exp(-B/T^{1/4}) \tag{8}$$

where A and B are constants and B is given by

$$B = 2.1[\alpha^3/k_{\rm B}N(E_{\rm F})]^{1/4} \tag{8a}$$

where $N(E_F)$ is the density of states at the Fermi level. On the other hand, Greaves [29] indicated that in the low-temperature region the conductivity can be expressed as

$$\sigma T^{1/2} = C \exp(-B/T^{1/4}) \tag{9}$$

where C is a constant and B is the same constant given in equation (8a).

Over a temperature range from about 550 to 380 K, the conductivities of G4-G8 obey equation (9). Figure 5 shows a plot of $\ln(\sigma T^{1/2})$ versus $1/T^{1/4}$ for the glasses G4-G8. There are two linear regions giving two *B*-values for each glass. The $N(E_F)$ -values can be obtained from equation (8*a*) by using the value of the wavefunction decay constant α . The values of $N(E_F)$ (table 3) differ slightly in the two regions. These values are reasonable for localized states [5, 12]. The lower slope in the high-temperature region is in agreement with the predictions of Triberis and Friedman [30]. Through application of the percolation theory, they predicted that the value of *B* should be lower in the high-temperature region, which is the case in the present study.

The temperature dependence of the DC conductivity in such glasses was also investigated by Holstein [31]. In his model, the conductivity is expressed as

$$\sigma = \frac{\pi e^2 J^2}{Rhk_{\rm B}T} \left(\frac{\pi}{Wk_{\rm B}T} \frac{\sinh(h\nu_{\rm o}/2k_{\rm B}T)}{h\nu_{\rm o}/2k_{\rm B}T} \right)^{1/2} \exp\left(-\frac{W+GW_{\rm p}}{2k_{\rm B}T}\right)$$
(10)

where

$$G = \frac{\tanh(h\nu_{\rm o}/2k_{\rm B}T)}{h\nu_{\rm o}/2k_{\rm B}T}$$
(10a)



Figure 5. Dependence of $\ln(\sigma T^{1/2})$ on $T^{-1/4}$ for the glasses G4–G6 and G8.

h is Planck's constant and J represents the overlap integral. The nature of hopping is related to the value of J by the following relation:

$$J \begin{cases} > \\ < \end{cases} (k_{\rm B}TW_{\rm H}/\pi)^{1/4} (h\nu_{\rm o}/\pi)^{1/2} \qquad \text{for } \begin{cases} \text{adiabatic hopping} \\ \text{non-adiabatic hopping} \end{cases}$$
(11)

with the condition for the formation of small polaron being $J \leq W_H/3$. From equation (10) the overlap integral value J can be calculated. J is found to be about 0.002 eV for G4 and about 0.02 eV for G8 and it ranges between these values for G5–G7. On comparison of these values with the previous conditions, it can be concluded that the non-adiabatic hopping is most appropriate for describing the polaronic conduction in the glasses investigated. Similar conclusions were obtained for Fe₂O₃–Bi₂O₃ glasses [9].

For electrons in localized states, the hopping mobility $\mu_{\rm H}$ (hopping being to nearest-neighbour sites) is of the form [12]

$$\mu_{\rm H} = (\nu_{\rm o} e R^2 / k_{\rm B} T) \exp(-2\alpha R) \exp(-W / k_{\rm B} T). \tag{12}$$

Values of the hopping mobility $\mu_{H(523 \text{ K})}$ at 523 K are given in table 3. μ_{H} increases with increase in Fe₂O₃ content, i.e. with increase in N. This behaviour is in agreement with the conductivity increase (with Fe₂O₃) as can be predicted from figure 1.

The sudden decrease in W and $\ln \rho$ (figure 2) for Fe₂O₃ content greater than 15 mol% is probably connected with structural changes. Saleh and Gawish [6] observed a similar change in the AC activation energy for Fe₂O₃ content greater than 16 mol% in xFe₂O₃-30CaO- $(70 - x)B_2O_3$ glasses. Their x-ray investigations indicated the presence of a crystalline structure in the glasses containing more than 20 mol% Fe₂O₃. It is expected that the tendency for a crystalline structure to be formed may start at relatively lower concentrations of Fe_2O_3 and this would affect the electrical properties. In the present study the x-ray diffraction patterns show a tendency for the formation of a crystalline structure in G8 (15 mol% Fe_2O_3). The spectrum of G10 (20 mol% Fe_2O_3) indicates clearly the presence of crystalline Fe_2O_3 in the structure.

Saleh and Gawish [6] concluded that for glasses having less than 20 mol% Fe_2O_3 there is a hopping conduction mechanism. For glasses having 20–30 mol% Fe_2O_3 (which are glass-crystal composites) they proposed a trap-limited band conduction mechanism together with a hopping conduction mechanism. This proposition can be considered, in the present study, for conduction in glasses having more than 15 mol% Fe_2O_3 .

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