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Study of rigidity of semiconducting vanadate glasses and its importance in use of coatings

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Abstract. **The elastic moduli of some multicomponent vanadate based glasses were analysed in terms of the bond compression model by some physical parameters such as, the density, average stretching force constant and average atomic ring size. These parameters were calculated for all the glass series and for all the glass composition to estimate the rigidity of these glasses. The results showed that the average force constant and the elastic moduli of these glasses are sensitive to the decrease in PbO content. This behaviour was attributed to the increase in the molar volume and the role of different modifiers. These parameters along with the coordination number of the glasses affect the glass transition temperature. The correlation between the elastic moduli and thermal properties of these samples showed that $0{\cdot}25MoO_3$–$0.25PbO$–$0{\cdot}5V_2O_5$ glass is the most rigid and has an applicable glass transition temperature for coating.**

Keywords. **Vanadate glasses; molar volume; elastic properties.**

1. Introduction

Understanding of the physical properties of semiconducting glasses has increased considerably following recognition of their potential applications for optical and memory switching devices and electrochemical batteries (Wells 1975; Ghosh 1988; Livage *et al* 1990; Montani *et al* 1992; Rao 2002). Semiconducting glasses can be fabricated from transition metal oxides (e.g. V_2O_5, PbO, MoO_3…) which constitute a class of non-conventional network formers (Baiocchi *et al* 1982; Josso and Calvarin 1986; Sidkey *et al* 2002). Due to the unique structural chemistry of crystalline oxides such as PbO and V_2O_5 (Calestani *et al* 1985), their counterpart lead vanadate glasses have wide glass-forming region. These glasses have been studied using electron spin resonance (ESR), magnetic susceptibility, thermodynamic measurements and DTA (Baiocchi *et al* 1982; Momo *et al* 1982; Calestani *et al* 1985; Josso and Calvarin 1986; Agostinelli *et al* 1986; Mandal and Ghosh 1993). Also, lead- and molybdenum-containing glasses were reported to be potential candidates for practical applications as amorphous semiconductors, for waste storage, infrared transmission components, thermal and mechanical sensors, reflecting windows or as catalysts for petroleum hydrotreatment (Dimitriev *et al* 1981, 1983a,b; Dimitriev and Mihailova 1990; Dimitrov and Dimitrov 1990; Dumbaugh and Lapp 1992; Iordanova *et al* 1994; Dimitrov 1995; Hayakawa *et al* 1995; Qiu *et al* 1997; Bahgat *et al* 1999; El Mallawany 2002; Mouhsine *et al* 2003; Hand *et al* 2005; Ramesh and Sastry 2006; Pisarska *et al* 2008). The semiconducting properties of these glasses arises from the transfer of charge carriers (electronic or polaronic) from lower-valence states (V^{4+}) to higher-valence states (V^{5+}) of the vanadium ions. IR spectral studies on a large number of these glasses revealed the existence of two different forms of distribution of metal ions in relation to vanadium–oxygen polyhedra, where Pb^{2+} occupies an interstitial position (Mandal and Ghosh 1990).

Studies of the elastic moduli of the vanadate glasses give considerable information about their structure, since the elastic moduli are directly related to the interatomic forces and potentials, i.e. they are determined by the rigidity of the structure (Pauling 1940; Bridge *et al* 1983; Bridge and Higazy 1986; Paul *et al* 1999; Saddeek *et al* 2009a, b). In literature, there are few studies on ternary and quaternary vanadate glass systems such as V_2O_5–BaO–ZnO or La_2O_3 and Fe_2O_3 are added to PbO–V_2O_5 glass system (Kawamoto *et al* 1979, 1980; Music *et al* 1994).

The purpose of this study is to analyse quantitatively the elastic moduli and hence the rigidity of series of multicomponent vanadate based glasses (Bridge and Higazy 1986; Paul *et al* 1999; Saddeek *et al* 2009) in terms of the bond compression model (Bridge *et al* 1983; Bridge and

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Higazy 1986). The used parameters are the bulk modulus, cross-link density, the stretching force constant and the average atomic ring size of these glasses. A correlation of such elastic moduli to the thermal properties of these glasses (Paul *et al* 1999; Saddeek *et al* 2009) will be achieved to select the most applicable glass for coating.

2. Theoretical analysis

The structure of oxide glasses can be described in terms of the bond compression model which was first introduced by Bridge *et al* (1983) for the study of single-component glass, was later developed for multicomponent glasses (Bridge *et al* 1986). The model is based on the observation that the measured bulk modulus K_e of oxide glasses is always markedly smaller than K_{bc}, the bulk modulus calculated under the assumption that each covalent bond experiences the linear contraction of the whole sample. Obviously, a sizable part of the volume change is due to a distortion of the network. In order to quantify the treatment of this effect, the model makes several assumptions:

(i) The elastic moduli only depend on both the 'connectivity' of the network (number of bonds per cation) and the average force constant f. The calculated bulk modulus (K_{bc}) according to this model can be estimated according to the relations:

$$K_{bc} = \left(\frac{\sum_i x n_b r_a^2 f}{9} \right) \text{(Gpa)}, \quad (1)$$

$$f = 5{\cdot}28N \left[\frac{X_a X_b}{r^2} \right]^{3/4} + 30 \ (\text{Nm}^{-1}), \quad (2)$$

where x is the mole fraction of the component oxide, n_b is the number of network bonds per unit volume, r_a is the bond length, N is the bond order (1, 2 or 3), X_a and X_b are the electronegativities of the anion and cation respectively. Accordingly, the average bond stretching-force constant F for a three-dimensional polycomponent oxide glasses can be estimated (Bridge *et al* 1983).

(ii) The ratio between the calculated elastic moduli and the experimental one (K_{bc}/K_e) is assumed to be directly proportional to the atomic ring size (ℓ). The atomic ring size is defined as the ring perimeter (number of bond times × bond length divided by π) and is given by the relation:

$$\ell = \left[0{\cdot}0106 \frac{f}{K_e} \right]^{0{\cdot}26} \text{(nm)}. \quad (3)$$

Therefore, a value of $K_{bc}/K_e \gg 1$ indicates a relatively open (i.e. large ringed) three-dimensional network with ring size tending to increase with K_{bc}/K_e and network bond bending (non-network compression) processes predominate when the glasses are subjected to bulk compression. The authors (Bridge *et al* 1983) found that the values of K_{bc}/K_e are 3·05, 3·08, 4·39 and 6·62 for pure SiO_2, P_2O_5, and GeO_2 and As_2O_3 glasses, respectively. The conformable atomic ring size of these glasses are 0·58, 0·567, 0·631 and 0·722 nm, respectively (Bridge *et al* 1983).

3. Results and discussion

The quantitative analysis of the literature data (Paul *et al* 1999; Saddeek *et al* 2009a, b) in terms of the bond compression model was done using the set of parameters listed in table 1. The set was adapted to the crystal structure of each oxide of the studied glass series (Wells 1975). These parameters are used in calculating the average stretching force constant f, the number of network bonds per unit volume n_b, the bulk modulus K_{bc}, the average atomic ring size ℓ; and the ratio K_{bc}/K_e. In table 1, one has two entries for the coordination number of V_2O_5 depending on the atomic composition, viz. $N = 5$ and 6, respectively (Music *et al* 1994; Saddeek *et al* 2009a, b).

The determination of the average cross-link density was based on the computation of both the coordination number of the constituent oxides. Also, it was found that the average force constant (f) is governed by the reciprocal of the bond length. Previously (Bridge *et al* 1983) it has been argued that the high value of the ratio $K_{bc}/K_e > 3$ indicates that network bond bending (or non-network bond compression) processes predominate when these materials are subjected to bulk compression. Thus, analysis of the computed elastic moduli according to the bond-compression model may be achieved for these glass series (Paul *et al* 1999; Saddeek *et al* 2009a, b) as follows.

3.1 *Analysis of elastic moduli*

3.1a *V_2O_5–PbO series*: Increasing PbO content on the expense of V_2O_5 in binary lead vanadate glasses results in

Table 1. Coordination number (N), cation–anion bond length (r_a), first-order stretching force constant (f) and electronegativity of cation (X_a), of oxides included in this study.

Oxides	N	r_a (nm)	f (Nm^{-1})	X_a
PbO	4	0·244	117·03	2·33
V_2O_5	5, 6	0·203	203·22	1·63
Li_2O	6	0·210	183·57	0·98
MoO_3	6	0·202	206·25	2·16

Values of coordination number, bond length and electronegativity of cation were taken from Wells (1975) and Saddeek (2009).

Table 2. Molar volume (V_m), number of network bonds per unit volume (n_b), average cross-link density ($\bar{n}_c$), bond compression bulk modulus (K_{bc}), ratio (K_{bc}/K_e), Poisson's ratio (σ), average force constant (F), average ring diameter (ℓ), average coordination number (N) and glass transition temperature (T_g) of studied glasses.

Composition in mol fraction														
PbO	V_2O_5	Li_2O	MoO_3	$V_m \times 10^{-6}$ (m^3/mol)	$n_b \times 10^{28}$ (m^{-3})	$\bar{n}_c$	K_e (GPa)	K_{bc} (GPa)	σ	K_{bc}/K_e	F (Nm^{-1})	ℓ (nm)	N	T_g (K)
0·500	0·500	–	–	39·87	6·80	2·667	39·90	58·52	0·219	1·467	165	4·428	4·50	521
0·488	0·512	–	–	39·80	6·83	2·677	34·89	58·91	0·219	1·689	166	4·593	4·51	520
0·482	0·518	–	–	40·11	6·78	2·682	36·61	58·59	0·219	1·600	166	4·540	4·52	518
0·452	0·548	–	–	41·63	6·58	2·708	38·55	57·12	0·218	1·482	169	4·496	4·55	518
0·402	0·598	–	–	44·14	6·27	2·748	30·75	54·94	0·217	1·787	173	4·799	4·60	519
0·45	0·50	0·05	–	40·02	6·92	2·774	38·61	60·04	0·217	1·555	168	4·489	4·60	513
0·40	0·55	0·05	–	40·60	6·90	2·813	37·97	60·33	0·216	1·589	172	4·537	4·65	511
0·35	0·60	0·05	–	41·66	6·79	2·848	37·33	59·91	0·216	1·605	176	4·585	4·70	506
0·30	0·65	0·05	–	42·62	6·71	2·882	36·81	59·67	0·215	1·621	180	4·628	4·75	504
0·45	0·50	–	0·05	39·79	6·96	2·733	30·9	60·54	0·218	1·959	170	4·768	4·60	521
0·40	0·50	–	0·10	40·38	7·01	2·800	31·9	61·54	0·216	1·929	174	4·762	4·70	523
0·35	0·50	–	0·15	41·46	6·97	2·867	33·2	61·75	0·215	1·860	179	4·743	4·80	527
0·30	0·50	–	0·20	42·05	7·02	2·933	33·7	62·69	0·214	1·860	183	4·754	4·90	528
0·25	0·50	–	0·25	43·20	6·97	3·000	34·9	62·78	0·213	1·799	187	4·737	5·00	529

Compositions and values of T_g of series V_2O_5–PbO were according to Paul *et al* (1999); V_2O_5–PbO–Li_2O were according to Saddeek *et al* (2009) and V_2O_5–PbO–MoO_3 were according to Saddeek (2009).

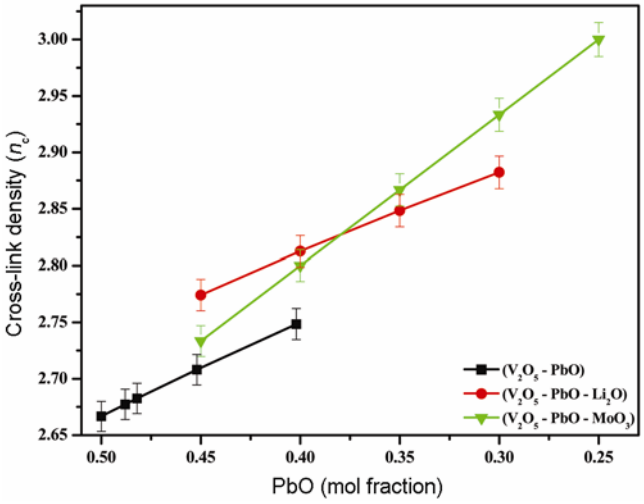

Figure 1. Compositional dependence of cross-link density on PbO content.

an increase in the molar volume, and the cross-link density along with decrease in the number of bonds per unit volume (n_b) as shown in figures 1–2 and as listed in table 2. The decrease in the number of bonds per unit volume is attributed to the increase in molar volume, i.e. a decrease in the compactness of the glass network structure confirming the creation of bridging oxygens BOs (Saddeek *et al* 2009a, b). The increase in the molar volume is attributed to the replacement of V_2O_5 has packing density $35{\cdot}4 \times 10^{-6}$ m^3 mol^{-1} by PbO has packing density 9×10^{-6} m^3 mol^{-1} (Inaba *et al* 2003). Moreover, the increase in cross-link density is attributed to the decrease in number of cations in the glass network structure and K_{bc} as shown in figure 3. Thus, the structure will be more linked and the network of these glasses becomes more compact as the electronegativity and cation field strength of PbO are higher than that of V_2O_5. The average atomic ring diameter as shown in figure 4 increases as a direct result which is attributed to the increase in both molar volume (i.e. interatomic spacing) and BOs. The suggestion by Bridge *et al* (1983) that the ratio K_{bc}/K_e (figure 5) is related to the average size of the rings may be accepted in fully polymerized glasses so; the relation between K_{bc}/K_e and the average ring size of this type of glasses is straightforward. As reported earlier, as the value of the ratio K_{bc}/K_e exceeds one, the connections between the structural units will be weaker and the structure becomes less stable. In addition, the decrease in the values of Poisson's ratio as shown in table 2 is mainly due to the increase in cross-link density while K_{bc} is found to increase and stretching force constant is found to decrease confirming that PbO has a pronounced bending effect on the behaviour of bond force constant.

3.1b *V_2O_5–Li_2O–PbO series*: According to table 2, the cross-link density increases and the K_{bc} decreases while the number of network bonds per unit volume decreases and the ring diameter increase as PbO content decreases. Also, the molar volume increases, oxygen molar volume decreased and the packing density (Saddeek *et al* 2009a, b) increases of these glasses with the decrease in PbO content. The increase in the average cross-link density is attributed to the replacement of PbO with V_2O_5 with higher coordination numbers which means the

increase in dimensionality of the glass network structure. On the other hand, the decrease in the number of network bonds per unit volume (figure 2) from $6{\cdot}92 \times 10^{28}$ to $6{\cdot}71 \times 10^{28}$ m^3 is attributed to the increase in molar volume and the decrease in oxygen molar volume which means the increase of BOs (Saddeek *et al* 2009a, b) with the decrease in PbO content. The computed values of these parameters are in a good agreement with the values given previously (Saddeek *et al* 2009a, b). The decrease in PbO content in the glass network results in the increased concentration of VO_5 and VO_6 structural units, as their coordination numbers are higher than PbO and hence lead to the increase in cross-link density. The decrease in K_{bc} with the decrease in PbO content is attributed to the replacement of PbO with higher electronegativity and higher cation field strength by V_2O_5 with lower electronegativity and cation field strength. Therefore, the increase in the values of the ratio K_{bc}/K_e and average ring diameter confirm the decreased rigidity of these glasses. Moreover, the decrease in the values Poisson's ratio confirm the change in the type of bonds from covalence to ionic, since the replacement of PbO (with higher cation field strength) by V_2O_5 would lead to the increase in the ionic cross-links and the increase in the average bond stretching force constant as seen in table 2 confirming the bending effect of PbO. Therefore, the decrease in the elastic moduli of these glasses along with the increase in ionic cross-links indicating some change in the type of bonding (Rajendran *et al* 2003).

3.1c *V_2O_5–PbO–MoO_3 series*: Incorporation of MoO_3 at the expense of PbO results in a markedly increase in

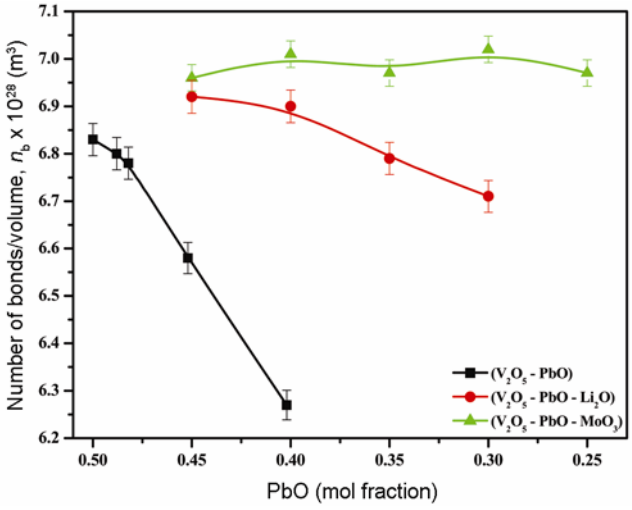

Figure 2. Compositional dependence of number of bonds per unit volume on PbO content.

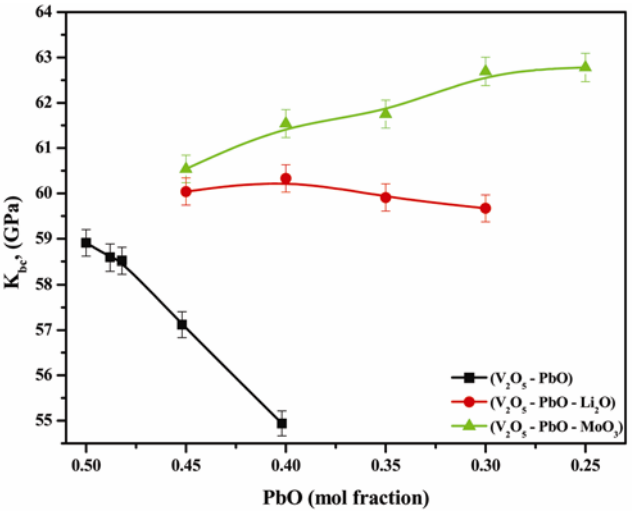

Figure 3. Compositional dependence of computed bulk modulus on PbO content.

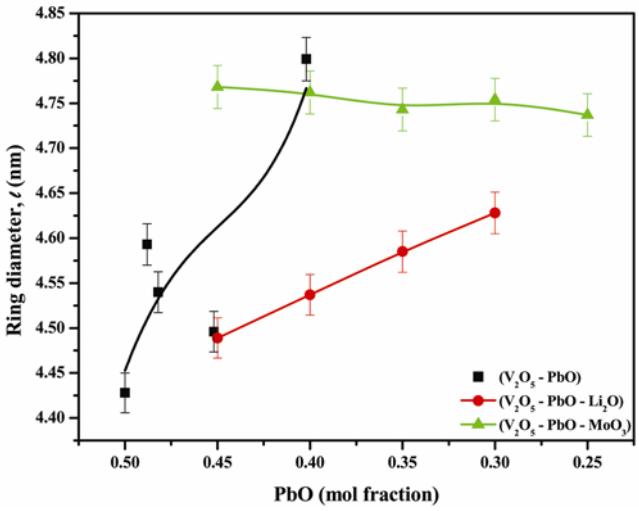

Figure 4. Compositional dependence of ring diameter on PbO content.

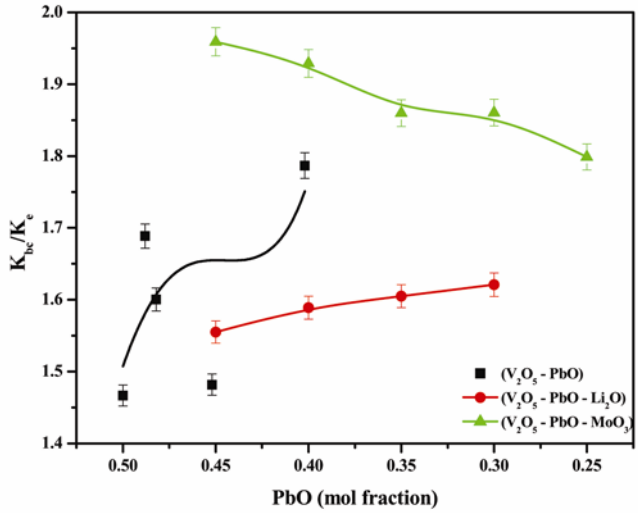

Figure 5. Compositional dependence of ratio K_{bc}/K_e on PbO content.

the molar volume and packing density (Saddeek *et al* 2009a, b). This incorporation will increase the average cross-link density and causes constancy in the total number of cations per unit glass formula unit (Bridge *et al* 1983; Bridge and Higazy 1986) and the number of network bonds per unit volume. In this type of glass, more polymerization will be created as the PbO content decreases, and as a result both the average ring diameter and K_{bc}/K_e will decrease and the relation between them is straightforward. The behaviour of the last parameters with the decrease in PbO content is attributed to the role of the structural units MoO_4 and MoO_6 (Saddeek *et al* 2009a, b) confirming the increased rigidity of the glass network structure due to the replacement of PbO with lower bond strength by MoO_3 with higher bond strength (Lide 2004). This behaviour was noticed (Gaafar *et al* 2009) in the experimental analysis of Bi_2O_3–Er_2O_3–PbO content glasses and was attributed to the role of PbO. Moreover, the decrease in the values Poisson's ratio confirm the increased rigidity and strength of cross-link density of these glasses through the change in the type of bonds which would lead to the increase in the cross-link density (i.e. increase in rigidity) and the increase in the average bond stretching force constant F as seen in table 2.

It is inferred that as PbO content decreases in the studied vanadate based glasses, it will increase molar volume, average cross-link density and force constant and decrease the number of bonds per unit volume, while K_{bc} is very sensitive to the rate of increase of the molar volume. Accordingly, the relation between the average ring diameter and K_{bc}/K_e depends on the polymerization or depolymerization of the vanadate network.

3.2 *Correlation between thermal properties and elastic moduli*

Recently, it was reported that the glass transition temperature T_g of glass-forming substances is a measure of structural packing of the network and is considered to be the temperature at which flow of the structure can be observed over long periods of time and the viscosity is just low enough (ca 10^{13} poise) to be measured accurately (Saddeek 2009). The glass transition temperature T_g can be related to some structural factors such as the average cross-link density and stretching force constant of each glass system. It was found that, T_g is an increasing function of both of $\bar{n}_c$ and f (El Mallawany *et al* 2002) with the decrease of PbO content as:

$$T_g = f(\bar{n}_c, f). \tag{4}$$

In PbO–V_2O_5 and Li_2O–PbO–V_2O_5 glasses, as the PbO content decreases, non-bridging oxygens in the vanadate network will be created, so the structure will be loosed which requires a smaller internal energy barrier for chain mobility, which is needed for the glass transition. Thus, T_g, K_{bc} and number of bonds per unit volume decrease while the ratio K_{bc}/K_e and the ring diameter increase which is a direct result of the increase of the molar volume. The decrease in K_{bc} may be related to changes in the coordination number Z of the constituent structural units of the glasses via the linear relationships: $\ln T_g = 2{\cdot}59Z + 0{\cdot}03$ and $\ln T_g = 2{\cdot}97Z - 0{\cdot}06$ for PbO–V_2O_5 and Li_2O–PbO–V_2O_5 glasses, respectively as shown in figure 6. Thus, the molar volume and number of bonds per unit volume have greater effects on T_g and the activation energy for glass transition (Saddeek 2009) than the bond strength (Saddeek *et al* 2009a, b).

In the while, for MoO_3–PbO–V_2O_5 glasses, as the PbO content decreases, more bridging oxygens in the vanadate network will be created, i.e. the structure will be strengthened which is attributed to the former role of MoO_3 and will require a larger internal energy barrier for chain mobility, which is needed for the glass transition causing an increase in the glass-transition temperature. Also, K_{bc}, the stretching force constant and the average cross link density will increase while the ratio K_{bc}/K_e and the ring diameter decrease which is a direct result of the increase of the bond strength. Therefore, the bond strength has greater effects on T_g and the activation energy for glass transition (Saddeek 2009) than the molar volume (Saddeek *et al* 2007). This increase may be related to changes in the coordination number Z of the constituent structural units of the glasses via the linear relationship: $\ln T_g = 2{\cdot}64Z + 0{\cdot}02$ as shown in figure 6.

The fascinating properties and wide range applications of V_2O_5 is especially interesting in thin film form because of the possibility of integration into micro-electronic circuitry and its application in optoelectronic devices. The properties of the V_2O_5 films are mainly dependent on the deposition technique and the deposition conditions

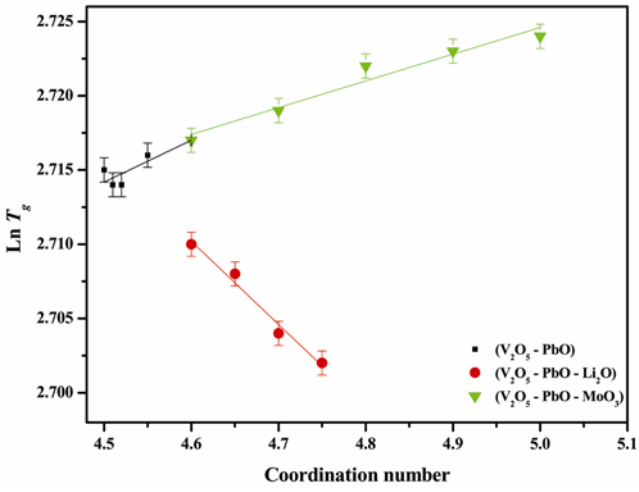

Figure 6. Dependence of glass transition temperature on coordination number.

such as vacuum, deposition temperature, etc. The deposition temperature in its turn depends on the melting temperature (T_m) of the material and consequently on its T_g. Another decisive parameter in the fabrication of the solar cell is the thermal stability which depends on the difference between T_g and T_m (Aly *et al* 2010, 2013). Thermal stable solar cell undergoes long times to the exposure of sun. On the basis of these arguments, Kumar *et al* (2008) coated amorphous glass substrates by homogeneous, uniform and amorphous V_2O_5 thin films by using vacuum evaporation technique at deposition temperature of 573 K. Deposition temperature is found to have a great impact on the optical and structural properties of these films. Therefore, addition of PbO and MoO_3 to V_2O_5 based glasses will reinforce such coating or thin film.

Thus, it is inferred from the former analysis that, the glass has the composition $0{\cdot}25MoO_3$–$0{\cdot}25PbO$–$0{\cdot}5V_2O_5$ is more rigid one and its glass transition temperature is suitable for thermal evaporation for the use as a thin film and coating for the fabrication of inexpensive solar cells.

4. Conclusions

In PbO–V_2O_5 and Li_2O–PbO–V_2O_5 glasses, as the PbO content decreases, non-bridging oxygens in the vanadate network will be created due to the increase in the molar volume, so the structure will be loosed and K_{bc} decreases. This decrease may be related to the changes in the coordination number Z of the constituent structural units of the glasses which is attributed to the effects of the molar volume and number of bonds per unit volume on T_g and the activation energy for glass transition than the bond strength.

While in MoO_3–PbO–V_2O_5 glasses, as the PbO content decreases, more bridging oxygens in the vanadate network will be created, i.e. the structure will be strengthened which is attributed to the former role of MoO_3. Thus, K_{bc}, the stretching force constant and the average cross-link density will increase while the ratio K_{bc}/K_e and the ring diameter decrease which is a direct result of the increase of the bond strength. Therefore, the bond strength has greater effects on T_g and the activation energy for glass transition than the molar volume.

The glass has the composition $0{\cdot}25MoO_3$–$0{\cdot}25PbO$–$0{\cdot}5V_2O_5$ is more rigid one and its glass transition temperature is suitable for thermal evaporation for the use as a thin film and coating.

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