

## Property–composition relationships for potentially bioactive glasses

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The compositional dependence of thermal expansion,  $\alpha$  (in  $10^{-6} \text{ K}^{-1}$ ), transformation temperature,  $T_g$  (in  $^{\circ}\text{C}$ ), dilatometric softening point,  $M_g$  (in  $^{\circ}\text{C}$ ), and viscosity,  $\eta$  (in dPa s), was determined for low-silica glasses in the system  $\text{Na}_2\text{O}-\text{K}_2\text{O}-\text{MgO}-\text{CaO}-\text{B}_2\text{O}_3-\text{P}_2\text{O}_5-\text{SiO}_2$ . The following phenomenological equations were established:

$$\alpha = 3.625 + 0.345 p_{\text{Na}_2\text{O}} + 0.266 p_{\text{K}_2\text{O}} + 0.098 p_{\text{CaO}} + 0.064 p_{\text{P}_2\text{O}_5},$$

$$T_g = 635.9 - 5.26 p_{\text{Na}_2\text{O}} + 3.18 p_{\text{K}_2\text{O}} - 3.16 p_{\text{MgO}} - 0.47 p_{\text{Na}_2\text{O}} \cdot p_{\text{K}_2\text{O}} - 0.12 p_{\text{K}_2\text{O}} \cdot p_{\text{CaO}},$$

$$M_g = 790.6 - 17.83 p_{\text{Na}_2\text{O}} - 5.79 p_{\text{K}_2\text{O}} + 0.28 p_{\text{Na}_2\text{O}}^2 - 0.39 p_{\text{MgO}}^2$$

where  $p_i$  is oxide content in wt%. The viscosity was modelled to fit an Arrhenius-type equation,  $\lg \eta = A + B/T$ . Giving  $\eta$  in dPa s, the compositional dependence of  $A$  and  $B$  were found as

$$A = -55.707 + 0.597 p_{\text{Na}_2\text{O}} + 0.597 p_{\text{K}_2\text{O}} + 0.604 p_{\text{MgO}} + 0.516 p_{\text{CaO}} + 0.429 p_{\text{P}_2\text{O}_5} + 0.387 p_{\text{SiO}_2},$$

$$B = -91.463 p_{\text{Na}_2\text{O}} + 162.861 p_{\text{CaO}} + 485.020 p_{\text{B}_2\text{O}_3} + 249.200 p_{\text{P}_2\text{O}_5} + 285.212 p_{\text{SiO}_2}.$$

### Beziehungen zwischen Eigenschaften und Zusammensetzung bei bioaktiven Gläsern

Der Einfluß der chemischen Zusammensetzung auf den Wärmeausdehnungskoeffizienten,  $\alpha$  (in  $10^{-6} \text{ K}^{-1}$ ), auf die Transformations-temperatur,  $T_g$  (in  $^{\circ}\text{C}$ ), auf den Erweichungspunkt,  $M_g$  (in  $^{\circ}\text{C}$ ), und auf die Viskosität,  $\eta$  (in dPa s), wurde für Gläser mit niedrigem Siliciumdioxidgehalt im System  $\text{Na}_2\text{O}-\text{K}_2\text{O}-\text{MgO}-\text{CaO}-\text{B}_2\text{O}_3-\text{P}_2\text{O}_5-\text{SiO}_2$  bestimmt. Die folgenden phänomenologischen Gleichungen wurden abgeleitet:

$$\alpha = 3.625 + 0.345 p_{\text{Na}_2\text{O}} + 0.266 p_{\text{K}_2\text{O}} + 0.968 p_{\text{CaO}} + 0.064 p_{\text{P}_2\text{O}_5},$$

$$T_g = 635.9 - 5.26 p_{\text{Na}_2\text{O}} + 3.18 p_{\text{K}_2\text{O}} - 3.16 p_{\text{MgO}} - 0.47 p_{\text{Na}_2\text{O}} \cdot p_{\text{K}_2\text{O}} - 0.12 p_{\text{K}_2\text{O}} \cdot p_{\text{CaO}},$$

$$M_g = 790.6 - 17.83 p_{\text{Na}_2\text{O}} - 5.79 p_{\text{K}_2\text{O}} + 0.28 p_{\text{Na}_2\text{O}}^2 - 0.39 p_{\text{MgO}}^2$$

wobei  $p_i$  für den Oxidgehalt in Gewichtsprozent steht. Die Viskosität wurde in eine Form gebracht, die einer Gleichung vom Arrhenius-Typ entsprach:  $\lg \eta = A + B/T$ . Für  $A$  und  $B$  wurden mit  $\eta$  in dPa s und in Abhängigkeit von der Zusammensetzung ermittelt:

$$A = -55.707 + 0.597 p_{\text{Na}_2\text{O}} + 0.597 p_{\text{K}_2\text{O}} + 0.604 p_{\text{MgO}} + 0.516 p_{\text{CaO}} + 0.429 p_{\text{P}_2\text{O}_5} + 0.387 p_{\text{SiO}_2},$$

$$B = -91.463 p_{\text{Na}_2\text{O}} + 162.861 p_{\text{CaO}} + 485.020 p_{\text{B}_2\text{O}_3} + 249.200 p_{\text{P}_2\text{O}_5} + 285.212 p_{\text{SiO}_2}.$$

### 1. Introduction

There is an increased interest in the possibility of cutting down the number of experiments, when designing glasses with specific properties. This interest is accentuated for properties which involve in-vivo experiments. On the other hand, many properties vary smoothly, even if rapidly, with composition and simple models are in general use in which the concentration of each oxide constituent is multiplied by a coefficient and all the terms are summed. An excellent review of available models has been compiled by Volf [1], but most of these concern ordinary commercial glasses. Only recently

attempts have been made to model also glasses in the composition range where bioactivity can be expected [2 to 4]. A bioactive glass is here defined as a glass to which bone cells attach by forming a chemical bond [2]. In general, glasses show bioactivity if the silica content is below 60 wt% [2 and 3], but the bioactivity decreases with increasing durability and is inhibited by trivalent ions like  $\text{Al}^{3+}$  [3]. The present study investigates physical properties in the low-silica part of the system  $\text{Na}_2\text{O}-\text{K}_2\text{O}-\text{MgO}-\text{CaO}-\text{B}_2\text{O}_3-\text{P}_2\text{O}_5-\text{SiO}_2$ . The property–composition relationships were determined in terms of thermal expansion, transformation temperature, dilatometric softening point and an approximation of viscosity. Good models are required for developing vitreous enamels for coating steel pro-

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Table 1. Composition by synthesis of the investigated glasses in wt%

glass no.	Na <sub>2</sub> O	K <sub>2</sub> O	MgO	CaO	B <sub>2</sub> O <sub>3</sub>	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>
1	10	5	0	15	0	0	70
2	15	0	2	15	0	0	68
3	0	15	5	10	0	3	67
4	10	5	0	15	3	3	64
5	15	0	5	10	0	6	64
6	5	10	2	20	0	0	63
7	5	10	2	15	3	6	59
8	10	5	5	15	3	3	59
9	15	0	5	20	3	0	57
10	20	10	5	10	0	0	55
11	20	10	2	10	3	3	52
12	20	10	5	10	3	0	52
13	25	5	2	10	3	3	52
14	15	15	2	15	3	0	50
15	20	10	5	20	0	3	42
16	25	5	2	20	0	6	42
17	20	10	5	20	0	6	39
18	15	15	2	20	3	6	39

theses with a layer able to form a firm chemical bond to bone.

## 2. Experimental design

Generating sufficient data to establish the properties for a seven-component system by classic methods would require hundreds of compositions to be investigated. Therefore, the authors used a computer search method for a minimum-correlated stochastic design of experiments [5]. The method, mentioned in [5], was developed by L. Nyström, but based on a suggestion by Himmelblau [6]. The experimental plan was designed to give as much information as possible to be used for a phenomenological modelling of the property–composition relationships.

Eighteen glasses with compositions given in table 1 were investigated. In order to get safe models for bioactive glasses, the compositional range was extended outside the region of bioactivity. Correlations between the compositions generated can be represented by means of a correlation matrix for the whole system. Values close to zero are preferred if the effect of each component is to be established independently of the others. But values close to 0.3 are still sufficient to avoid elimination of wrong variables in the regression analysis procedure. The correlation matrix is given in table 2. Because the oxides total 100%, SiO<sub>2</sub> was chosen as a dependent variable and omitted from statistical analysis. Thus, cross correlations involving silica are of no concern.

## 3. Experimental methods

The glasses were prepared from pure dry materials, melted in a platinum crucible in an electric furnace and homogenized by crushing and remelting. The melts were

cast to rods in a graphite mould and annealed for 30 min slightly above the estimated temperature  $T_g$  (470 to 580 °C) and slowly cooled to room temperature according to a scheme suggested by Redston and Stanworth [7]. The rods were cut and ground to a length of 25 mm and a cross section of (5 by 5) mm<sup>2</sup>. The thermal expansion was determined by a Dilatronic I dilatometer from Theta Industries Inc. The reproducibility of an expansion measurement was about 2%. At least two parallel measurements were made for each glass. If the difference between two measurements exceeded 2%, the measurement was repeated twice and the two extremes skipped. Also, if the expansion curve indicated poor annealing, the sample was reannealed. From the dilatometric curve three different properties were extracted, the thermal expansion,  $\alpha$ , the transformation point,  $T_g$ , and the dilatometric softening point,  $M_g$ .

Further, an attempt was made to estimate the viscosity–temperature relationship according to a method described by Scholze [8]. The method is suitable as a first approximation when the number of experiments is large. It is also suitable for compositions containing components which have a high vapour pressure or which corrode platinum. According to the method certain deformation points observed in a high-temperature microscope (Leitz Wetzlar GmbH, Wetzlar (Germany)) can be related to the viscosity. The microscope consists of an electrically heated horizontal tube furnace in which a cylindrical sample (2 by 2) mm<sup>2</sup> made from crushed glass is placed on a ceramic substrate. The deformation of the sample is observed when the furnace is heated. Scholze defined four points that can be reproducibly identified, i.e. the sintering point,  $SP$ , defined as the point where the sample starts shrinking, the minimum base line point,  $MBL$ , where the sample already is a soft glass, but the wetting of the substrate is negligible. Further, the point when the sample forms a semisphere

Table 2. Correlation matrix showing cross correlation between oxides in experiment plan

	Na <sub>2</sub> O	K <sub>2</sub> O	MgO	CaO	B <sub>2</sub> O <sub>3</sub>	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>
Na <sub>2</sub> O	1.000						
K <sub>2</sub> O	-0.191	1.000					
MgO	0.117	0.029	1.000				
CaO	0.000	0.000	-0.114	1.000			
B <sub>2</sub> O <sub>3</sub>	0.041	0.116	-0.185	-0.136	1.000		
P <sub>2</sub> O <sub>5</sub>	0.096	0.080	0.047	0.168	0.000	1.000	
SiO <sub>2</sub>	-0.654	-0.402	-0.215	-0.420	-0.149	-0.435	1.000

which he denoted by *HKP*, and the point when the sample has floated out to a given height he called flow point (*FP*). The microscope was calibrated with a commercial container glass with known viscosity–temperature behaviour between  $T_g$  and the temperature where  $\eta = 10^2$  dPa s. Giving the viscosity  $\eta$  in dPa s, the points were identified as follows:  $\eta = 10^{10.0 \pm 0.3}$  at *SP*,  $\eta = 10^{6.1 \pm 0.3}$  at *MBL*,  $\eta = 10^{4.2 \pm 0.3}$  at *HKP* and finally  $\eta = 10^{3.4 \pm 0.3}$  at *FP*. The latter two values are lower than found by Scholze [8], who reported  $\eta = 10^{4.55}$  to  $10^{5.0}$  at *MBL* and  $\eta = 10^{4.1}$  to  $10^{4.3}$  at *FP*, respectively. Further, establishing *MBL* is strongly influenced by the wetting of the ceramic substrate. In particular high potash contents lowered the surface tension too much for any accurate determination. Also the two high-viscosity points, *SP* and *MBL*, were difficult to establish. On the other hand,  $T_g$  and  $M_g$  can be reproducibly determined within a few degrees from the thermal expansion and these points were taken to represent the high-viscosity region. For the calibration glass these two points were found to correspond to  $\eta = 10^{13.3}$  and  $10^{11.8}$  dPa s, respectively. It was therefore decided to use the two dilatometric points and *HKP* and *FP* for modelling the viscosity–composition relationships.

#### 4. Results

The linear thermal expansion between 20 and 300 °C and the temperatures at the four chosen viscosity points are given in table 3. Expansion values are means of two determinations. The scattering in the expansion measurements never exceeded 2%, while  $T_g$  and  $M_g$  could be determined within about  $\pm 5$  K. The two viscosity values at *HKP* and *FP* are arithmetic averages of three determinations. The scatter was much greater, but less than  $\pm 20$  K corresponding to less than  $\pm 0.25$  viscosity units. The scatter was of the same order as obtained when calibrating the instrument.

The compositional dependence of the coefficient of expansion,  $\alpha$  (in  $10^{-6}$  K<sup>-1</sup>), between 20 and 300 °C was tested at a 95 % significance level. The model

$$\alpha = 3.625 + 0.345 p_{\text{Na}_2\text{O}} + 0.266 p_{\text{K}_2\text{O}} + 0.098 p_{\text{CaO}} + 0.064 p_{\text{P}_2\text{O}_5} \quad (1)$$

where  $p_i$ , is the amount of the  $i$ -th oxide in wt%, has a regression coefficient  $R^2 = 99.67\%$  and an estimated standard deviation of  $0.17 \cdot 10^{-6}$  K<sup>-1</sup>. The degrees of freedom, i.e. the number of experiments used for testing the model, were 13. The goodness of the fit was estimated by an F-test and the total F-value was found to be 982.

The compositional dependence of the transformation temperature,  $T_g$  (in °C), was also tested at a 95 % level and could be described by

$$T_g = 635.9 - 5.26 p_{\text{Na}_2\text{O}} + 3.18 p_{\text{K}_2\text{O}} - 3.16 p_{\text{MgO}} - 0.47 p_{\text{Na}_2\text{O}} \cdot p_{\text{K}_2\text{O}} - 0.12 p_{\text{K}_2\text{O}} \cdot p_{\text{CaO}} \quad (2)$$

Here are: the regression coefficient  $R^2 = 99.35\%$ , the estimated standard deviation 6.8 K, the degrees of freedom 12 and the total F-value 368.

The compositional dependence of the dilatometric softening point,  $M_g$  (in °C), was

$$M_g = 790.6 - 17.83 p_{\text{Na}_2\text{O}} - 5.79 p_{\text{K}_2\text{O}} + 0.28 p_{\text{Na}_2\text{O}}^2 - 0.39 p_{\text{MgO}}^2 \quad (3)$$

The model was tested at a 95 % significance level. The regression coefficient was  $R^2 = 99.5\%$  and the estimated standard deviation 5.9 K. The degrees of freedom were 13 and the total F-value was 643.

In testing the viscosity models a good enough description was obtained with an Arrhenius-type equation

$$\lg \eta = A + B/T \quad (4)$$

A Vogel-Fulcher-Tammann relationship was therefore never tested, in particular as it had required non-linear regression analysis. For viscosity (in dPas) the compositional dependence of  $A$  and  $B$  was found to be

$$A = -55.707 + 0.597 p_{\text{Na}_2\text{O}} + 0.597 p_{\text{K}_2\text{O}} + 0.604 p_{\text{MgO}} + 0.516 p_{\text{CaO}} + 0.429 p_{\text{P}_2\text{O}_5} + 0.387 p_{\text{SiO}_2} \quad (5)$$

$$B = -91.463 p_{\text{Na}_2\text{O}} + 162.861 p_{\text{CaO}} + 485.020 p_{\text{B}_2\text{O}_3} + 249.200 p_{\text{P}_2\text{O}_5} + 285.212 p_{\text{SiO}_2} \quad (6)$$

Table 3. Experimental and calculated thermal expansion between 20 and 300 °C, transformation temperature, dilatometric softening point, high-temperature-microscopic semisphere and flow point. The calculated *HKP* and *FP* are obtained from equations (4 to 6).

glass no.	$\alpha$ in $10^{-6} \text{ K}^{-1}$		$T_g$ in °C		$M_g$ in °C		<i>HKP</i> in °C		<i>FP</i> in °C	
	experi- mental	equation (1)	experi- mental	equation (2)	experi- mental	equation (3)	experi- mental	equation (4)	experi- mental	equation (4)
1	9.88	9.89	570	566	614	611	1065	1060	1145	1130
2	10.12	10.28	557	551	588	584	1055	1032	1100	1102
3	8.63	8.80	652	649	695	694	1095	1108	1170	1183
4	10.02	10.08	565	566	604	611	955	1008	1060	1071
5	10.14	10.17	531	541	577	576	1010	1024	1115	1094
6	10.20	9.99	597	587	642	649	1135	1108	1160	1185
7	9.84	9.87	581	593	661	649	1040	1038	1185	1105
8	10.28	10.08	550	551	593	602	870	1006	1005	1073
9	10.81	10.77	545	541	581	576	1065	987	1085	1055
10	14.21	14.17	444	440	486	478	945	963	1000	1047
11	14.76	14.36	449	449	488	486	930	911	955	982
12	14.14	14.17	441	440	475	478	950	902	995	975
13	14.66	14.75	447	449	487	489	920	879	930	947
14	14.10	14.27	464	465	500	498	975	952	1010	1028
15	15.23	15.35	435	428	478	478	990	1000	1005	1099
16	15.85	15.93	443	443	488	489	1085	968	1095	1059
17	15.60	15.54	422	428	478	478	1010	1005	1030	1105
18	15.15	15.15	453	456	494	498	995	974	1010	1057

Table 4. Compositional validity range in wt% for equations (1 to 3, 5 and 6)

oxide	range of composition in wt%
Na <sub>2</sub> O + K <sub>2</sub> O	15 to 30
K <sub>2</sub> O	0 to 15
MgO + CaO	10 to 25
MgO	0 to 5
B <sub>2</sub> O <sub>3</sub>	0 to 3
P <sub>2</sub> O <sub>5</sub>	0 to 6

where  $p_i$  is given in wt%. Giving viscosity in dPa s, for equation (4) the regression coefficient was  $R^2 = 98.74$  at a 95% significance level and the estimated standard deviation for  $\lg \eta = 0.53$ . The degrees of freedom were 60 and the total F-value was 439. The validity range of all models is given in table 4.

## 5. Discussion

### 5.1 Thermal expansion

The greatest recorded difference between an experimental and a predicted coefficient of expansion was  $0.44 \cdot 10^{-6} \text{ K}^{-1}$ , but in general the difference was less than 0.1. The problem is, however, not the reproducibility nor the modelling, but the question of how to interpret the dilatometric recording. There is in every dilatometer at low temperatures a difference between actual sample temperature and thermocouple temperature. For instance, as a rule  $\alpha$  between room temperature and 300 °C is about 10% smaller than between 70 and 300 °C. Further, in literature the linear expansion is sometimes reported between room temperature and

300 °C, sometimes between room temperature and 400 °C. For the set of glasses in this investigation, the values of  $\alpha_{20/400}$  were about 5% larger than those of  $\alpha_{20/300}$ .

One of the best methods published for estimating the linear expansion coefficient from composition was published by Appen [9] back in 1949, i.e. long before a detailed design of experiments by statistical analysis of big bodies of data were commonly used. Applying his model on the present set of compositions gave as a rule very good agreement with experiments. Appen's model yielded on average 0.7% higher values, but extremes as big as -4.6 and +4.2% were recorded, corresponding to about  $0.3 \cdot 10^{-6} \text{ K}^{-1}$ .

### 5.2 Dilatometric points

The two dilatometric points,  $T_g$  and  $M_g$ , could be very accurately determined. Although the experimental plan showed very small cross correlations, in the final model several oxide terms were eliminated in the F-test due to cross correlations. The modelling also required second-degree compositional terms. However, the final models give very good estimates in the investigated region.

Andersson [4] has published a model for  $T_g$  based on DTA measurements on bioactive glasses. Although MgO and K<sub>2</sub>O are not considered in his model, a reasonable agreement is obtained between estimates by his model and by equation (2). As a rule the results are within the limit given for equation (2) and large deviations occur only for glasses outside Andersson's experimental silica range. In the same paper Andersson also gives a restricted model based on the soda content only. This model, however, is not applicable in the present experiments.

### 5.3 Viscosity

The determination of the viscosity–composition relationship was made by combining two different methods, the dilatometric points for the low-temperature region and the high-temperature microscope for the working-range region. The difference between experimental value and estimate according to equation (4) was fairly independent of the experimental method and on the average less than 0.3 units on the logarithmic scale. The accuracy of the model is, however, less, only about 1 unit. In the low-temperature region this corresponds to about  $\pm 50$ , in the working range to about  $\pm 100$  K. Although these values are rather large, the accuracy is not inferior to other comparable models [1].

When comparing the measured viscosities with viscosities calculated by the models developed by Lakatos [10 and 11] and Lyon [12], the agreement was acceptable only for the glasses with the highest silica content. At low silica contents the estimated low-temperature viscosities were wrong by orders of magnitude. Because Lakatos' and Lyon's models do not consider  $P_2O_5$ , they were tested only on a limited set of experiments, but even then it was evident that they gave poor estimates in the region for bioactive compositions, i.e. below about 60%  $SiO_2$ .

Recently Brink [13] has made an attempt to estimate the viscosity by using all four of Scholze's microscopic points. The discrepancy between her model and equations (5 and 6) is particularly great in the low-temperature region. Here the dilatometric determination seems to be superior to the high-temperature microscope. However, the present authors also obtained a slightly lower viscosity in the working range.

### 6. Conclusions

The aim of this investigation was to model properties important when enameling steel in order to get a surface to which bone cells can attach. Thus, the important viscosity region is at temperatures in and below the working range. The compositional dependence of thermal expansion, of transformation point, of dilatometric softening point and of viscosity of potentially bioactive glasses has been determined in the low-silica part of the system  $Na_2O-K_2O-MgO-CaO-B_2O_3-P_2O_5-SiO_2$

on eighteen glasses chosen by a minimum-correlated stochastic design of experiments. Acceptable relationships are developed for all properties investigated. The experimental range covers compositions also outside the range of bioactive glasses. Models for compositional dependence of bioactivity have, however, been treated elsewhere [2,3 and 14].

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