

DTA STUDY OF TEMPERATURE CHARACTERISTICS IN DEPENDANCE ON COMPOSITION FOR GLASSES IN THE $\text{CaO} - \text{MgO} - \text{Al}_2\text{O}_3 - \text{SiO}_2 - \text{B}_2\text{O}_3$ SYSTEM.

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ABSTRACT

The analytical expressions for the positions of characteristic temperature points on DTA-curves for glasses in the system $\text{CaO} - \text{MgO} - \text{Al}_2\text{O}_3 - \text{SiO}_2 - \text{B}_2\text{O}_3$ (CMASB) were obtained by multilinear regression analysis. The expressions for the dependence of individual characteristic temperature points on glass composition are valid in the region determined by standard composition of the commercial glass EUTAL altering concentrations of all the oxides up to - 4 mass %.

INTRODUCTION

As Yamamoto¹ showed, it is possible to give a physical interpretation for the characteristic points on DTA-curves of glasses. He suggested a method based on this proposal for determination of characteristic temperatures corresponding to some viscosity points. In the paper submitted the dependence of some point positions on DTA-curves upon the composition of system, which represents a base for the commercial glass EUTAL, is investigated.

MEASURING METHODS

The DTA-curves were measured by DTA-analyser NETZSCH 404. The heating rate was $10^\circ\text{C}/\text{min}$, registration sensibility $4 \mu\text{V}/\text{cm}$. 0.5 g of a sample was prepared as a powder having grain size less than 0.016 mm. As a reference material powdered $\alpha\text{-Al}_2\text{O}_3$ was used. The measurements were carried out in Pt-crucibles in open air-atmosphere.

RESULTS AND DISCUSSION

41 glass samples of the CMASB-system were investigated in this study. Compositions of the glasses obtained by chemical analysis are shown in table 1. In the difference of Yammamotos' work¹ four important points were observed on the DTA-curves: T_g - transition point, M_g - incipient deformation point, B_c - beginning of crystallization, T_{soft} - Littletons' softening point. Temperature of each important point was expressed as a function of composition by following regression relation

$$T(\bar{c}) = \sum_i \sum_j \sum_k \sum_l \sum_m A_{i,j,k,l,m} \cdot c_1^i c_2^j c_3^k c_4^l c_5^m$$

where $c_1 - c_5$ are concentrations of the oxides in mass percentage in the sequence: CaO, MgO, Al₂O₃, SiO₂, B₂O₃. Optimal regression functions were obtained by the least squares method including statistically important coefficients A only and a maximal value of Fischers' criterion (defined by the experimental to residual dispersion ratio) was required, too.

Values of the exponents i, j, k, l, m and the coefficients $A_{i,j,k,l,m}$ of regression functions for T_g , M_g , B_c and T_{soft} are presented in tabs. 2 - 5. In tab. 6 experimental values of the temperatures are compared with values calculated using the regression functions.

Results presented in tab. 6 enable us to suppose that the regression formulas suggested in this paper are suitable for calculating individual characteristic temperatures with a precision sufficient for practice. Since these are interpolation formulas it is obvious, that they can not be used to extrapolate outside the region of their validity, which is given by the concentration range of CMASB glasses studied.

Table 1.: Composition of the samples in mass %

Sample	%CaO	%MgO	%Al ₂ O ₃	%SiO ₂	%B ₂ O ₃
1	18.72	3.85	14.88	53.82	7.93
2	18.79	3.77	14.77	49.58	12.08
3	18.64	3.87	14.01	51.97	10.25
4	18.66	3.81	14.97	55.78	6.16
5	18.66	3.87	14.97	57.21	4.29
6	18.67	3.84	18.9	49.78	8.06
7	18.57	3.93	16.77	52.04	7.94

Table 1.: Continuation

Sample	%CaO	%MgO	%Al ₂ O ₃	%SiO ₂	%B ₂ O ₃
8	18.65	3.97	13.05	55.34	8.01
9	18.71	3.84	11.08	57.85	8.08
10	19.01	3.84	11.13	53.69	11.6
11	18.85	3.92	12.99	53.71	10.04
12	19.00	3.9	17.1	55.8	4.18
13	18.62	3.85	18.85	53.54	4.15
14	18.5	0.00	18.87	53.58	8.18
15	18.41	1.9	16.95	53.48	8.3
16	18.77	5.81	12.93	53.46	8.28
17	18.43	7.91	11.1	53.52	8.18
18	18.34	0.00	15.1	53.63	11.97
19	18.52	2.14	15.07	53.54	10.21
20	18.65	6.05	14.98	53.72	6.22
21	19.21	7.68	14.94	53.61	4.12
22	15.14	7.79	14.96	53.36	8.00
23	16.94	5.91	15.01	53.47	8.18
24	20.61	2.15	14.99	53.44	8.24
25	22.42	0.00	15.06	53.49	8.14
26	14.82	4.00	14.79	53.92	11.84
27	16.75	4.00	14.9	53.51	9.89
28	20.64	4.13	15.04	53.65	6.15
29	18.32	3.66	14.02	58.56	5.44
30	22.66	3.99	14.84	53.42	4.22
31	16.74	3.95	16.94	53.64	8.04
32	14.66	4.01	18.98	53.47	8.02
33	22.56	4.04	11.03	53.29	8.16
34	22.68	4.15	15.11	49.79	7.89
35	20.67	4.04	15.07	51.9	7.77
36	16.74	4.05	15.09	55.27	7.89
37	14.87	3.98	15.1	57.55	8.05
38	19.11	7.95	14.97	49.41	8.1
39	18.77	6.15	14.98	51.64	8.07
40	18.57	2.15	14.98	55.46	8.07
41	18.55	0.00	15.09	57.45	8.01

Table 2.:

Regression function for T_g

A(i,j,k,l,m)	i	j	k	l	m
+5.3307E+3	0	0	0	0	0
-1.0804E+2	0	0	0	0	1
+1.2695E+1	0	0	0	0	2
-1.3839E+2	0	0	0	1	0
+1.0410E+0	0	0	0	2	0
-8.1309E+1	1	0	0	0	0
+1.5467E+0	1	0	0	1	0
-5.0746E-1	0	0	0	0	3
+9.0757E-3	0	0	1	1	1

Table 3.:

Regression function for M_g

A(i,j,k,l,m)	i	j	k	l	m
-5.6332E+0	0	0	0	0	1
+1.6965E+2	0	0	1	0	0
-1.1533E+1	0	0	2	0	0
-2.6704E+0	0	1	0	0	0
+2.6771E-1	0	2	0	0	0
-2.0292E+0	1	0	0	0	0
+2.5963E-1	0	0	3	0	0

Table 4.:
regression function for B_c

A(i,j,k,l,m)	i	j	k	l	m
-8.8999E+2	0	0	0	0	0
+7.6006E+1	0	0	0	0	1
+3.9435E-1	0	0	0	2	0
+1.6352E+2	0	0	1	0	0
-4.9710E+0	0	0	1	0	1
-2.3556E+0	0	0	1	1	0

Table 5.:
Regression function for T_{soft}

A(i,j,k,l,m)	i	j	k	l	m
-1.0866E+3	0	0	0	0	0
+1.0765E+2	0	0	0	0	1
+2.3006E+1	0	0	0	1	0
+2.5108E+1	1	0	0	0	0
-2.2899E+0	1	0	0	0	1
-4.1473E-1	1	0	1	0	0
+1.3695E-1	0	3	0	0	0
+5.1033E-2	0	0	3	0	0
-5.9060E-2	0	0	1	1	1

Table 6.: Comparison measured vs. calculated values (truncated) [Pc]

Spl.	T_g^{exp}	T_g^{cal}	M_g^{exp}	M_g^{cal}	B_c^{exp}	B_c^{cal}	T_{soft}^{exp}	T_{soft}^{cal}
1	694	680	728	726	826	821	872	860
2	684	675	706	703	803	812	848	862
3	687	685	716	713	798	818	846	865
4	695	687	734	735	817	834	869	878
5	719	718	743	746	840	842	894	890
6	697	698	727	729	810	827	859	855
7	681	684	724	722	820	815	872	850
8	675	681	721	728	830	846	931	894
9	704	697	731	720	926	906	958	957
10	656	660	693	701	934	907	971	970
11	694	679	721	717	873	851	918	904
12	710	706	757	751	851	877	901	917
13	702	696	738	739	848	810	891	893
14	678	689	732	726	822	815	881	864
15	669	673	720	722	816	827	879	889
16	668	666	713	710	845	831	982	950
17	672	677	718	713	825	829	901	894
18	701	688	722	717	826	825	860	876
19	688	676	728	729	814	817	867	868
20	707	699	735	736	825	811	881	886

REFERENCES

- 1 A. Yamamoto, Proc. of the 1st International Conference on TA, Aberdeen 1965, McMillan Co., London 1965