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FEATURES OF THE CALCULATION OF THE LIQUIDUS TEMPERATURE OF SODIUM BOROSILICATE GLASSES IN THE PRESENCE OF Fe_2O_3 AND Cr_2O_3

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Abstract. This paper discusses the application of Kauzmann's rule for calculating the liquidus temperature of glasses in the Na_2O–B_2O_3–SiO_2 system. The dependence of the adjusted glass transition temperature (Tgr) on the structure of sodium borosilicate glass is established, and the influence of Fe_2O_3 and Cr_2O_3 additives on this parameter is investigated. For compositions prone to crystallization and aventurine formation, a regression equation is calculated to describe the dependence of Tgr on the chemical composition of the glasses. Additionally, a methodology for applying Kauzmann's rule to calculate the liquidus temperature of oxide glasses, considering the dilatometrically determined glass transition temperature (Tg), is proposed for the studied compositions. This allows the calculation of the liquidus temperature without the need for differential thermal analysis.

Keywords: glass, Kauzmann's rule, glass transition temperature, liquidus temperature, crystallization.

1. Introduction

The Na_2O–B_2O_3–SiO_2 system is one of the most extensively studied glass-forming systems, enabling it to serve as a basis for the production of chromium-containing and iron-containing aventurine glass-ceramic materials, as well as other glasses, glazes, and enamel coatings with specified properties.[1-8]

Sodium borosilicate glasses, over a wide composition range, are capable of maintaining their resistance to crystallization even after thermal treatment, making it possible to use them as a transparent base for producing glass materials of various functional purposes. The introduction of Fe_2O_3 or Cr_2O_3 oxides into the composition of such glasses allows for the predictable crystallization of hematite or eskolaite within the glass phase, resulting in a glass-ceramic material with the manifestation of the aventurine effect – a shimmering light.[7,9-11]

For most glasses, the ability to crystallize upon cooling the melt is an undesirable process, as it disrupts the conditions for glass production and damages its quality characteristics. However, for artificial aventurine materials (glasses and coatings), the ability of the glass melt to crystallize is a defining factor.

Aventurine formation, as a process of bulk crystallization in the glass melt, depends on the formation of crystallization centers and crystal growth. It can occur both during the cooling of the melt, *i.e.*, "top-down," and during the heating of the glass, *i.e.*, "bottom-up." Initially, nuclei – crystallization centers – form, and then crystals grow from them. The formation of crystallization centers can occur homogeneously due to local fluctuations in the structure, as well as heterogeneously in the presence of foreign impurities, gas bubbles, and existing contacts between phase boundaries, for example, "glass melt – air" or "glass melt – ceramics." For artificial aventurine materials based on sodium borosilicate glasses, both types of crystallization center formation, both homogeneous and heterogeneous, are typical.

In general, the dependence of the crystallization center formation rate (CCFR) and crystal growth rate (CGR) on the temperature of the glass is shown in Fig. 1.

The crystallization temperature range of glass melts is limited by the lower crystallization temperature T_2 and the upper crystallization temperature, which corresponds to the liquidus temperature of the melt (T liq).

A low CGR and a high CCFR, which can be observed at temperatures close to T_2, create conditions for the formation of a large number of small crystals. Such thermal treatment conditions are more suitable for the production of glass-ceramics.

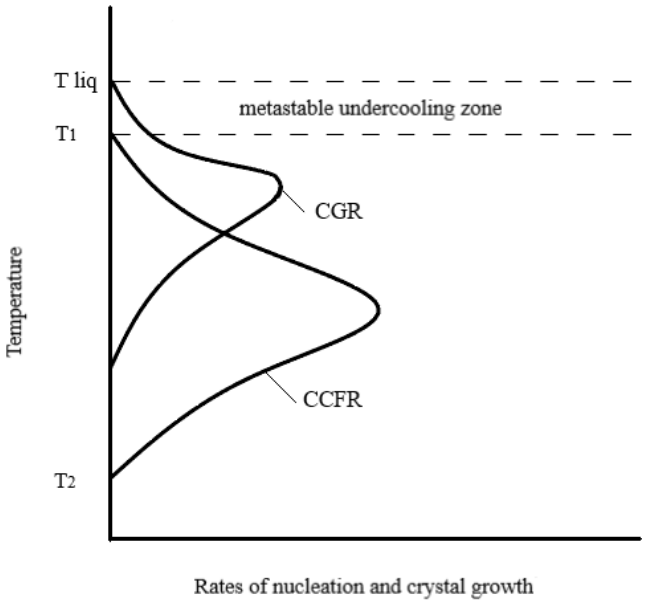


Fig. 1. Effect of temperature on the crystallization center formation rate (CCFR) and crystal growth rate (CGR) in the glass melt [7]

At high CGR and low CCFR, which can be observed at temperatures close to the liquidus temperature, the most likely result in the melt is the formation of a small number of large crystals. The process of forming a small number of large crystals occurs with the simultaneous dissolution of small crystals under conditions of low viscosity in the melt at temperatures close to the liquidus temperature. With slight adjustments and lowering of the temperature, these thermal treatment conditions can increase the number of formed crystals with sizes that lead to the manifestation of the decorative shimmering light effect.

It should be noted that, depending on the composition of the glass, during the cooling of the melt in the "top-down" mode within the temperature range Tliq – T_1, a metastable undercooling zone may be observed, where crystallization centers do not form. This can influence the choice of thermal treatment temperature for the melt.

An additional significant factor affecting the processes of crystallization center formation and crystal growth during the production of artificial aventurine, and also determining the firing temperature of glass coatings, is the viscosity of the glass. It is commonly assumed that the viscosity of glasses at the liquidus temperature is close to $10^{2.5}$ Pa·s[1].

It is known[1,12] that the viscosity of the glass melt during the formation of coatings on substrates made of different materials should be within the range of $10^{1.5}$–10^3 Pa·s, and should not exceed $10^{3.5}$ Pa·s.

It is also known[13] that for crystal growth during the production of aventurine glaze coatings, the viscosity of the glaze melt at the firing temperature should be 10^3 Pa·s.

Therefore, the formation of coatings can be carried out at a temperature slightly higher than the liquidus temperature of the glass, but the thermal treatment, during which nucleation and crystal growth occur, should be performed at a temperature slightly lower than the liquidus temperature. This corresponds to a melt viscosity value of 10^3 Pa·s and correlates with the CGR and CCFR dependencies shown in Fig. 1.

The influence of temperature on the viscosity of sodium borosilicate glass melts is well studied, based on data from the SciGlass electronic database and research by scientists related to experimental-statistical modeling of this dependence.[11,14] According to these data, the viscosity of sodium borosilicate melts, including iron-containing glasses, changes from $10^{2.5}$ Pa·s to 10^3 Pa·s when the temperature decreases by about 50 degrees.

Thus, knowing the liquidus temperature of sodium borosilicate glasses in the presence of aventurine-forming oxides (Fe_2O_3, Cr_2O_3) is an important and essential factor, allowing for a reasoned selection of the firing temperature of coatings and the thermal treatment temperature, during which bulk crystallization may occur.

According to Kauzmann's rule,[15] when cooling melts at a rate of 10–100 K/s, the adjusted glass transition temperature (Tgr) of oxide glasses is calculated as the ratio of the glass transition temperature to the liquidus temperature and is approximately:

$$\mathrm{Tgr} = \frac{\mathrm{Tg}}{\mathrm{T\,liq}} \approx \frac{2}{3} = 0.67\ .$$

The formula for calculating the adjusted glass transition temperature (Tgr) using Kauzmann's rule can be used to determine the liquidus temperature (T liq) of glasses. Kauzmann's rule has been confirmed by the results of differential thermal analysis for oxide glasses, as well as for sulfide and fluorosulfide glasses.[16]

The objective of this study is to validate the applicability of Kauzmann's rule to experimental sodium borosilicate glasses and to develop an algorithm for calculating their liquidus temperature, a key parameter for predicting optimal thermal treatment conditions during aventurine crystallization.

2. Experimental

2.1. Materials

The study was conducted on glasses from the Na_2O–B_2O_3–SiO_2, Na_2O–B_2O_3–SiO_2–Fe_2O_3 and Na_2O–B_2O_3–SiO_2–Cr_2O_3 systems with a Na_2O/B_2O_3 ratio of 1.0 and 1.5. The compositional domain of the experimental glasses was

selected based on an analysis of the phase diagram of the $Na_2O–B_2O_3–SiO_2$ system,[17] which includes several low-temperature eutectic points:

a) Within the elementary triangle $Na_2O·B_2O_3–SiO_2–Na_2O·2SiO_2$ (covering the primary crystallization fields of $Na_2O·B_2O_3$, SiO_2, and $Na_2O·2SiO_2$), melt crystallization terminates at a ternary eutectic point (Na_2O – 27.0 wt.%, B_2O_3 – 24.0 wt.%, SiO_2 – 49.0 wt.%) with a temperature of 510 ± 10 °C;

b) Within the elementary triangle $Na_2O·2SiO_2–Na_2O·SiO_2–Na_2O·B_2O_3$ (covering the primary crystallization fields of $Na_2O·2SiO_2$, $Na_2O·SiO_2$, and $Na_2O·B_2O_3$), crystallization ends at a binary maximum (Na_2O – 32.5 wt.%, B_2O_3 – 17.0 wt.%, SiO_2 – 50.5 wt.%) with a temperature of 640 ± 10 °C.

The presence of these eutectic points, along with the potential existence of the ternary compound $Na_2O·B_2O_3·2SiO_2$ with relatively low melting temperatures within the relevant region of the $Na_2O–B_2O_3–SiO_2$ phase diagram selected for this study, supports the assumption that low-melting glasses suitable as base materials for aventurine can be obtained.

The experimental glasses were melted in corundum crucibles with a volume of 100 mL in a laboratory electric furnace with silicon carbide heaters for 30 minutes at temperatures between 1100–1350ºC. The temperature in the furnace was measured using a Pt–Pt-Rh thermocouple. The readiness of the glass was determined visually by thread breakage.

For preparing the raw batches of the experimental glasses, finely ground quartz sand was used, along with raw materials of grades "chemically pure" and "analytical grade": boric acid (H_3BO_3), sodium carbonate (Na_2CO_3), iron (III) oxide (Fe_2O_3), and chromium (III) oxide (Cr_2O_3).

2.2. Methods

Differential thermal analysis (DTA) of the glasses was carried out in corundum crucibles on a MOM Q-1500D derivatograph of F. Paulik, H. Paulik, H. Erdei system in the temperature range of 20–1000ºC with a heating rate of 10ºC/min. The reference substance was α-Al_2O_3.

Dilatometric measurements of the glass transition temperature (Tg) were performed on a quartz dilatometer DKV-4. The measurements were conducted on glass samples with dimensions of 4×4×50 mm.

High-temperature studies of the structural transformations of the experimental glasses during heating and subsequent cooling, the processes of nucleation of crystallization centers and further crystal growth, as well as the photographing of these processes, were carried out using an MBS-10 microscope (magnification ×32). The studies were performed on ceramic samples with a thickness of 3 mm and a diameter of 10 mm, covered with a 2 mm layer of experimental glass. The glass was applied to the test sample using the enamel technology.

3. Results and Discussion

The analysis of the derivatograms and consideration of the structural features of the experimental sodium borosilicate glasses[10] allowed us to establish a fundamental characteristic of the application of Kauzmann's rule for glasses in the $Na_2O–B_2O_3–SiO_2$ system. Since boron tetrahedra in sodium borosilicate glasses cannot form a common bond with each other and have intermediate bridging and non-bridging bonds with boron triangles and silicon tetrahedra, the glass network can contain boron triangle groups alongside boron and silicon tetrahedra. It is known that boron triangles, when joined at their vertices, can form boroxol rings, which correspond to the structure of B_2O_3. These boron groupings are characterized by a low melting temperature (≈ 450 ºC) and influence the liquidus temperature of the sodium borosilicate melt[1].

The calculation of the adjusted glass transition temperature (Tgr) in this work is presented using the example of experimental glass 6 (mol.%: 30 – Na_2O; 20 – B_2O_3; 50 – SiO_2), taking into account the glass transition temperature and liquidus temperature, which were determined by the DTA method (Fig. 2).

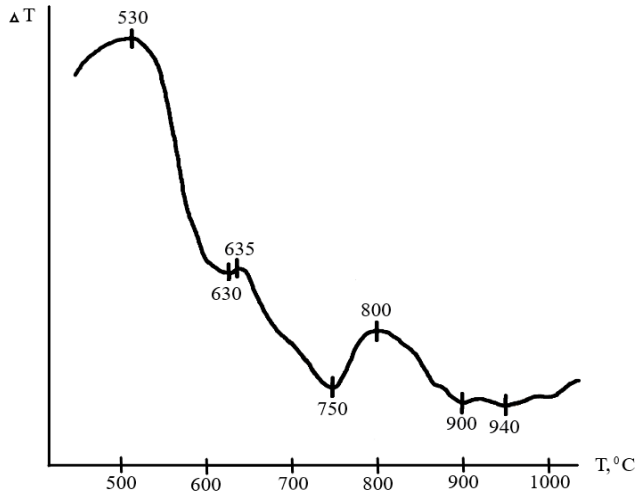


Fig. 2. Derivatogram of glass 6

The glass transition temperature (Tg) of the experimental glass is 530°C.

The liquidus temperature (T liq) of the experimental glass is 940°C.

The adjusted glass transition temperature (Tgr) of the experimental glass is:

$$\frac{\text{Tg}}{\text{T liq}} = \frac{530}{940} = 0.56$$

According to the "classical" conditions of Kauzmann's rule, the calculated value of (Tgr) 0.56 does not correspond to the theoretical value of ≈ 2/3 = 0.67. The discrepancy between the calculated and theoretical adjusted glass transition temperature can be explained by the factor of the presence of silicon and boron groupings in tetrahedral coordination, as well as boron in triple coordination in the glass structure. Therefore, the structure exhibits differentiation, manifested by two endo-effects at 630°C and 750°C.

The liquidus temperature, calculated separately for the borate and silicate components of the glass, according to Kauzmann's rule, is 791°C and 947°C, respectively:

$$\text{T liq (B)} = \frac{530}{0.67} = 791; \text{T liq (Si)} = \frac{635}{0.67} = 947\ .$$

Since the actual liquidus temperature of the experimental glass (940°C), determined by differential thermal analysis, correlates with the calculated liquidus temperature of the silicate component of the glass (947°C), it should be concluded that the liquidus temperature of sodium borosilicate glasses is determined by the characteristics of the higher-melting silicate component of the structure. Therefore, the glass transition temperature (Tg) and liquidus temperature (T liq) for the experimental sodium borosilicate glass are 530°C and 940°C, respectively. This is why the adjusted glass transition temperature (Tgr) of the experimental glass is 0.56.

Thus, using differential thermal analysis, glasses from the $Na_2O–B_2O_3–SiO_2$ system with a composition of (mol.%) 20–30 Na_2O, 16–25 B_2O_3, and 50–60 SiO_2, as well as their derived compositions with the separate addition of Fe_2O_3 (ranging from 5 to 12.5 mol.%) and Cr_2O_3 (ranging from 0.5 to 2 mol.%), were studied. The concentrations of Fe_2O_3 and Cr_2O_3 additives used in this study were selected based on an analysis of known iron- and chromium-containing aventurine glasses and glazes.[7,9,11]

Within the investigated concentration range of Fe_2O_3 and Cr_2O_3, it was found that the effect of Fe_2O_3 on $Na_2O–B_2O_3–SiO_2$ glasses is considerably more significant compared to Cr_2O_3 and leads to a decrease in the Tgr value within the experimental composition range from 0.56-0.54 to 0.44-0.48. The effect of Cr_2O_3, within the concentration range of 2 mol.%, almost does not influence the Tgr value of the base sodium borosilicate glasses and causes only a slight decrease in the Tgr value from 0.56-0.54 to 0.55-0.53.

Based on the results of the studies, mathematical modeling of the dependence of the adjusted glass transition temperature (Tgr) on the chemical composition of the experimental glasses was performed using the additivity method in the form of equation:[18]

$$p = \sum i\ xi \qquad (1)$$

where p is the calculated value of Tgr of the glass; i is the additive coefficient of the influence of a specific oxide on the Tgr value of the glass; xi is the oxide content in the glass, mol.%.

Regression equations have been developed that describe the dependence of the adjusted glass transition temperature (Tgr) of the experimental glasses on their composition (mol %) using first-order additivity Eqs (2, 3). The accuracy of the calculations based on the proposed equations is confirmed by the pairwise correlation coefficients between the calculated and experimental values of the reduced glass transition temperature (Tgr) for the experimental glasses (0.992; 0.995), which are typically considered as a criterion for assessing the adequacy of mathematical models of partial oxide properties in multicomponent glass systems.[19]

Fe_2O_3: $Tgr = 0{,}0070 \cdot Na_2O + 0{,}0057 \cdot B_2O_3 + 0{,}0049 \cdot SiO_2 - 0{,}0019 \cdot Fe_2O_3$ (2)

Cr_2O_3: $Tgr = 0{,}0070 \cdot Na_2O + 0{,}0048 \cdot B_2O_3 + 0{,}0050 \cdot SiO_2 + 0{,}0005 \cdot Cr_2O_3$ (3)

The practical significance of the obtained results for the calculation of the adjusted glass transition temperature (Tgr) for the experimental glasses of the $Na_2O–B_2O_3–SiO_2–Fe_2O_3$ and $Na_2O–B_2O_3–SiO_2–Cr_2O_3$ systems, and their further use in the development of new coating compositions, is as follows:

For any glass composition within the studied range, taken as the base for further coating development, dilatometric determination of the glass transition temperature (Tg) is performed;

– For this glass, the adjusted glass transition temperature (Tgr) is calculated according to the developed regression equation, and the liquidus temperature of the glass is calculated using Kaufmann's rule:

$$T_{liq} = \frac{Tg}{Tgr};$$

– Based on the calculated liquidus temperature, the firing temperature of the coatings and the thermal processing temperature for crystallization and adventuring formation are predicted.

Example of practical application of the method:

The composition of the glass 6Fe10 (mol.%) – 27 Na_2O, 18 B_2O_3, 45 SiO_2, 10 Fe_2O_3.

The softening temperature (Tg) according to dilatometric measurements is 456°C.

The derived softening temperature (Tgr):

$$Tgr = 0{,}0070 \cdot 27 + 0{,}0057 \cdot 18 + 0{,}0049 \cdot 45 - 0{,}0019 \cdot 10 = 0{,}49.$$

The liquidus temperature (T liq):

$$T_{liq} = \frac{456}{0.49} = 930°C\ .$$

The correctness of the calculated parameters is confirmed by comparison with the data from differential thermal analysis of the 6Fe10 glass (Fig. 3).

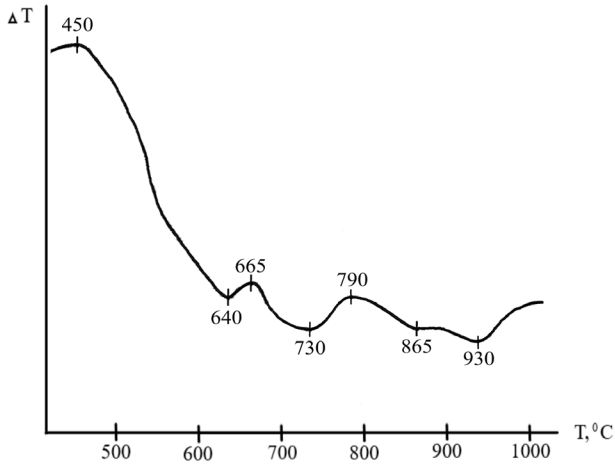


Fig. 3. Derivatogram of glass composition 6Fe10 (mol.%): 27 Na_2O, 18 B_2O_3, 45 SiO_2, 10 Fe_2O_3.

Thus, the work demonstrates the possibility of determining the liquidus temperature (T liq) of glasses using two independent methods: from the results of differential thermal analysis and by calculations according to the Kaufmann rule. This temperature specifically determines the firing temperature of coatings based on this glass. Undoubtedly, the actual firing temperature of coatings may slightly differ from the liquidus temperature of the base glass due to the addition of ingredients to the slip that adjust its rheological properties and influence its physicochemical and operational characteristics.

Experimental studies of the structural transformation processes of experimental glass 6Fe10 during heating, and thus confirmation of the correctness of the chosen liquidus temperature (930°C) and thermal treatment temperature (880°C), were conducted using a high-temperature microscope. To ensure the transition of the glass into the liquid state, the heating was carried out up to 950°C with a 15-minute hold.

At a temperature of 950°C, a complete transition of the experimental glass to the liquid state was confirmed, accompanied by a degassing process – the release of air bubbles, which was almost completely finished within 15 minutes (Fig. 4).

Further thermal treatment of the glass melt was carried out after partial cooling to a temperature of 880°C, which is 50°C lower than the calculated liquidus temperature. Isothermal holding was performed for 30 minutes.

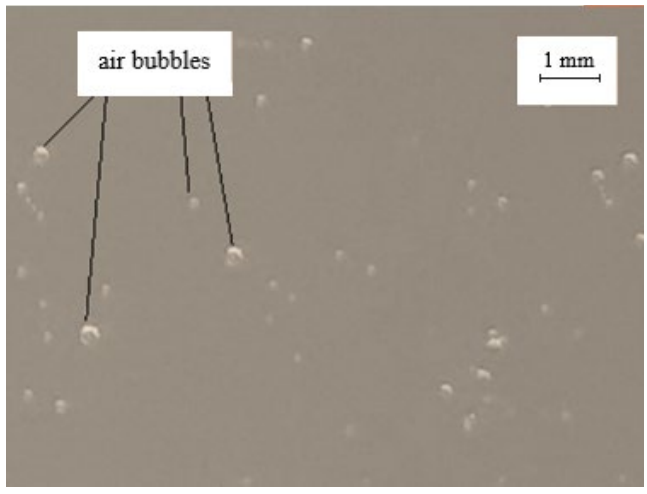
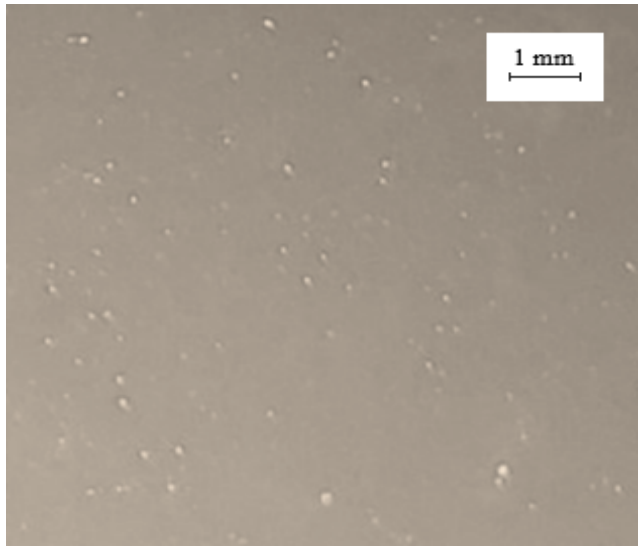


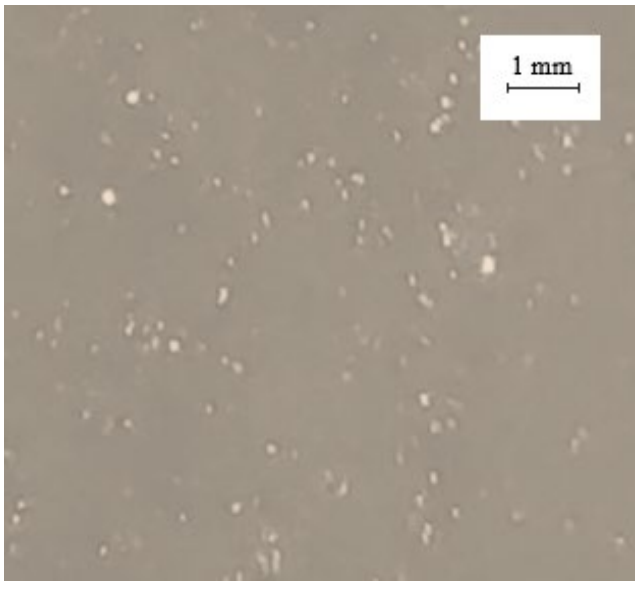
Fig. 4. Photo of the surface structure of experimental glass 6Fe10 after holding at 950°C for 15 minutes

After the first 10 minutes of holding at 880°C, crystallization centers began to form in the glass melt, and the size of the crystalline phase increased partially (Fig. 5a). Further holding at the thermal treatment temperature for 30 minutes (Fig. 5b) was accompanied by crystal growth without a significant increase in the number of crystallization centers, which is characteristic of the region within the SRK curve (Fig. 1). The formed aventurine phase consists of hematite crystals with an average size of 200 μm, which is fully consistent with the characteristic features of aventurine formation in iron-containing glasses and glazes.[7,9]



a

Fig. 5. Photo of the surface structure of experimental glass 6Fe10: a) – after holding at 880°C for 10 minutes



b

Fig. 5. Continuation**.** Photo of the surface structure of experimental glass 6Fe10: b) – after holding at 880°C for 30 minutes

The study at the selected temperatures and thermal treatment durations allowed obtaining the expected results in both the quality of the formed surface and the achieved volume crystallization of the experimental glass. However, it should be noted that the tendency of glasses to undergo volume crystallization and adventure formation is determined not only by the thermal treatment conditions but also by the glass composition and melt structure. The ability of Fe^{3+} ions to exist in both tetrahedral and octahedral coordination directly influences the possibility of crystallization processes.

4. Conclusions

As a result of the conducted research using differential thermal analysis, the effect of additives of 5–12.5 mol.% Fe_2O_3 and 0.5–2 mol.% Cr_2O_3 on the adjusted glass transition temperature (Tgr) of glasses with the composition (mol.%) 20–30 Na_2O, 16–25 B_2O_3, 50–60 SiO_2 was established. The effectiveness of Kauzmann's rule for the experimental oxide glasses was also confirmed, and a methodology for calculating their liquidus temperature was proposed.

It was found that an increase in Fe_2O_3 content up to 12.5 mol.% leads to a decrease Tgr from the range of 0.56-0.54 to 0.44-0.48, while an increase in Cr_2O_3 content up to 2 mol.% has almost no effect on the Tgr value of the base sodium borosilicate glasses, which decreases from the range of 0.56-0.54 to 0.55-0.53.

The liquidus temperature of the glasses, which serves as a technological marker for the subsequent selection of the thermal treatment temperature for glasses and coatings, is proposed to be calculated using Kauzmann's rule with the use of the dilatometrically determined glass transition temperature (Tg) and the adjusted glass transition temperature (Tgr) calculated from the developed regression equations.

The paper experimentally confirms the accuracy of the calculations of the liquidus temperature (T_liq) and the selection of the thermal treatment temperature for the glass melt, which ensures the occurrence of crystallization and aventurine formation processes. This has further scientific and practical significance in the development of new compositions of synthetic aventurine materials.

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ОСОБЛИВОСТІ РОЗРАХУНКУ ТЕМПЕРАТУРИ ЛІКВІДУСУ НАТРІЙБОРОСИЛІКАТНИХ СТЕКОЛ У ПРИСУТНОСТІ Fe_2O_3 ТА Cr_2O_3

Анотація. *У роботі розглянута особливість застосування правила Каузмана для розрахунку температури ліквідусу для стекол системи Na_2O–B_2O_3–SiO_2, встановлена залежність приведеної температури склування (Tgr) дослідних стекол від структури натрійборосилікатного скла, а також досліджено вплив добавок Fe_2O_3 та Cr_2O_3 на показник Tgr. Для області складів, схильних до кристалізації й авантюриноутворення, розраховано рівняння регресії залежності приведеної температури склування (Tgr) від хімічного складу стекол. Для стекол дослідженої області складів запропоновано методику застосування правила Каузмана за проведення розрахунків температури ліквідусу дослідних оксидних стекол з урахуванням дилатометрично визначеного значення температури склування (Tg). Ці розрахунки дають змогу визначити температуру ліквідусу стекол без проведення диференційно-термічного аналізу.*