

CONCENTRATION CHANGES IN THE SURFACE GLASS LAYER DURING INTERACTION WITH AgNO₃ MELT AND A MIXTURE OF AgNO₃ AND NaNO₃

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The content of silver and its concentration distribution in the surface layer of a multicomponent glass was determined after contact with AgNO₃ melt or a mixture of AgNO₃ and NaNO₃. Apart from the concentration profile of silver the measurements also included the concentration profile of sodium ions in the glass. The data obtained were used in the determination of the mean silver diffusion coefficient value and its dependence on the concentration of silver in the glass.

INTRODUCTION

Interaction of the glass surface with the melt of an inorganic salt is a complex phenomenon involving several processes. In the system silicate glass — AgNO₃ melt the following processes are usually considered:

- a) phase boundary reaction of the type Ag⁺ (melt) ⇌ Me⁺ (glass),
- b) diffusion of Ag⁺ ions through the melt towards the phase boundary,
- c) diffusion of Ag⁺ ions through the glass in the direction from the phase boundary,
- d) diffusion of alkali ions through the glass towards the phase boundary,
- e) diffusion of alkali ions liberated from the glass through the AgNO₃ melt in the direction from the phase boundary.

Under certain conditions it is necessary to take into account further complicating processes related to the behaviour of silver ion in the glass lattice (reduction, nucleation and crystallization of silver in glass).

As a result of these processes the concentration of silver ions gradually increases and that of alkali ions in the glass surface layer decreases. The changes in concentration bring about changes in the properties of glass. For instance silver ions in the surface layer are known to modify the optical, electrical and mechanical properties of the basic glass [1—3]. The relationship between the changes in properties and the concentration parameters has not so far been completely elucidated. However, one may assume that the degree of changes in the properties depends above all on the content and concentration distribution of silver and on the form in which silver is present in the structural lattice of glass. With some glasses the changes in concentration in the surface layer were determined following a certain time of contact with the melt of silver salts [4]. The data published so far, however, are rather incomplete and do not allow to determine explicitly the conditions under which surface layers of the required content and concentration profile of silver in glass are formed. The present study has the aim of expanding the knowledge in the given direction and of determining the decisive parameters for the preparation of layers with a defined silver content and a defined silver concentration distribution.

EXPERIMENTAL

The experiments were carried out with the „Unihost“ technical glass containing 68.8 SiO₂, 2.7 Al₂O₃, 4.0 MgO, 6.2 CaO, 0.8 K₂O, 17.3 Na₂O (% by wt.) in the form of a tube of approx. 11 mm outer diameter and 1—1.5 mm wall thickness. The tubes were cut into lengths of approx. 40 cm and adjusted to test-tube type specimens by sealing at one end. Silver nitrate of A.R. purity also in mixture with sodium nitrate of the same grade was used in the preparation of the melt.

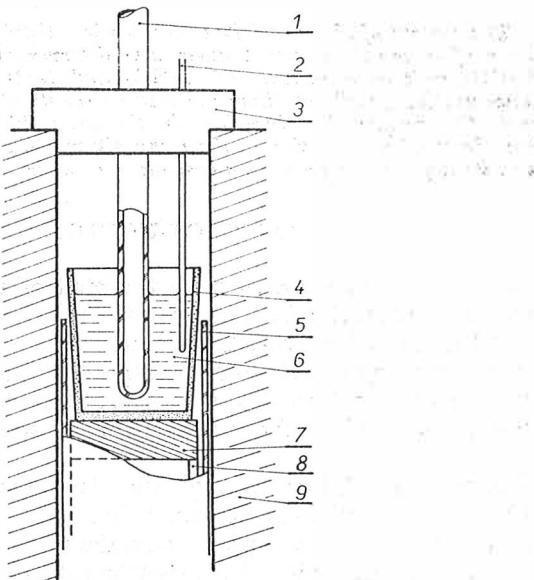


Fig. 1. Apparatus for thermal exposure of specimens; 1 — glass specimen, 2 — thermocouple, 3 — metal flange, 4 — corundum crucible, 5 — quartz glass tube, 6 — melt, 7 — corundum lid, 8 — corundum tube, 9 — electric furnace.

The glass specimens were thermally exposed using an apparatus the schematic diagram of which is shown in Fig. 1. The salt bath temperature was measured by an Au — 40 Pd/Pt — 10 Rh thermocouple placed inside a quartz glass protective tube. The temperature control was ensured by the TRS 114 controller with a Pt/Pt — 10 Rh. thermocouple. The salt bath was heated up to the working temperature of 350 °C and kept at this temperature within the limits of ± 5 °C. Before exposure the glass specimen was rinsed in distilled water and ethanol. A corundum crucible filled with silver nitrate or the AgNO₃ + NaNO₃ mixture was placed into the furnace and heated up to the working temperature. The thermal exposure of the specimen was started by immersing it into the salt bath. After the required period of time the specimen was removed from the bath, cooled in air, and rinsed with distilled water and ethanol. The specimen section which had been immersed in the melt was cut off and used for determining the Ag content and its distribution throughout the glass surface layer.

The silver content in the glass was determined by etching off the surface layer in 6 % HF. The resulting solution was transferred into a platinum dish and evaporated dry. The dry residue was dissolved in 8—9 ml mixture of concentrated HF and HClO_4 (5 : 3.5), re-evaporated dry and transferred quantitatively into a 100 ml capacity measuring flask. The Ag content in the solution was determined by potentiometric titration with 0.001—0.005 % KBr solution. The completeness of etching off of the surface layer was checked by repeated etching of the specimen.

The silver concentration distribution in the surface layer was determined by gradual etching of thin layers and with the use of an electron microprobe. The etching procedure has been described in the previous paragraph. In view of the low Ag content in the thin etched off layers the Ag content was determined by atomic absorption spectrophotometry. The electron microprobe samples were prepared by casting pieces of the exposed glass into a polyester resin, by grinding it on a wheel with strongly bound abrasive, and final polishing on cerium oxide. Following vacuum deposition of a carbon layer the sample was analyzed on an electron microprobe. The surface concentration of Ag in density units was determined by means of a standard (pure Ag) while respecting the matrix effects in the computer calculation.

THE RESULTS AND DISCUSSION

The content of silver and its concentration profile was determined on specimens thermally exposed in an AgNO_3 melt. The time dependence of silver content is shown in Fig. 2. The linear shape of the dependence of Ag content on the second root of time seems to indicate that the process in question is controlled by a diffusion process. According to the concept described in the Introduction one of the slow diffusion processes in the glassy phase may be

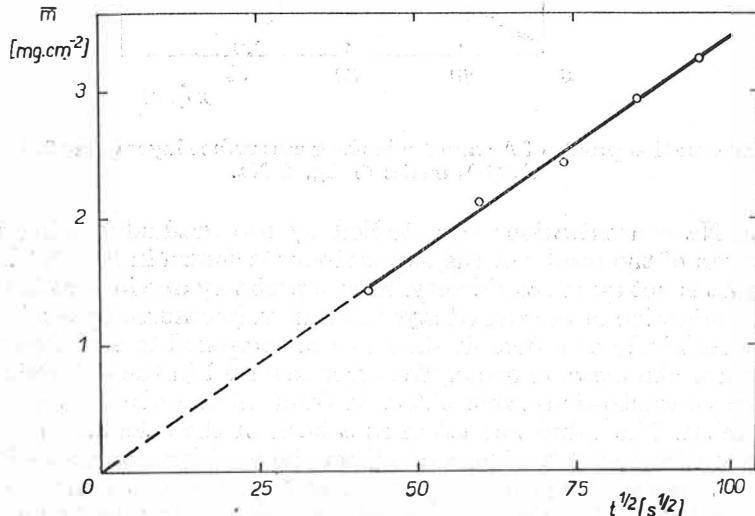


Fig. 2. Silver content in glass vs. second root of time (350 °C, AgNO_3 melt).

considered to act as the controlling process. More detailed information may be obtained from the concentration profiles of the individual ions. In view of their low content in the glass the potassium ions were not taken into consideration, although they obviously take part in the diffusion process to a very limited degree. Figs. 3—5 summarize the data on the distribution of sodium and silver ion concentrations in the glass surface layer obtained from the electron probe measurements. The thickness of the glass surface layer in which the concentration changes take place increases with time. The course

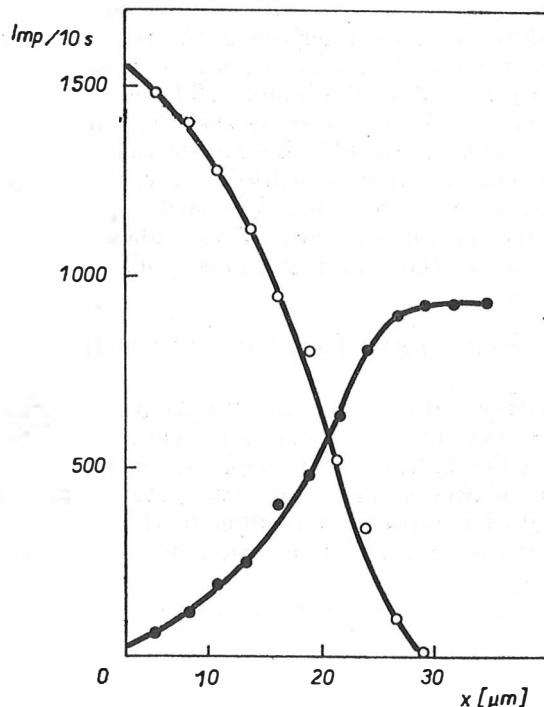


Fig. 3. Concentration profile of Ag and Na in the glass surface layer (time 0.5 hr, 350 °C, AgNO_3 melt); ○ Ag, ● Na.

of Ag and Na concentrations was studied by the gradual etching method. A comparison of the results of the two methods is shown in Fig. 6. The agreement of data is not quite satisfactory. This is probably due in part to the error in the determination of the etched layer thickness, (measured by a micrometer) and to the relatively considerable thickness as compared to the steps taken in the electron probe measurements. The error involved in the determination of the surface concentration value obtained from microprobe measurements is also significant. The value was taken as a basis in the calculation of density within the entire concentration range from the working concentration units (pulses) on the assumption of direct proportionality. A comparison of concentration profiles obtained from electron microprobe measurements on the one hand and from the etching on the other indicates that the surface concentration

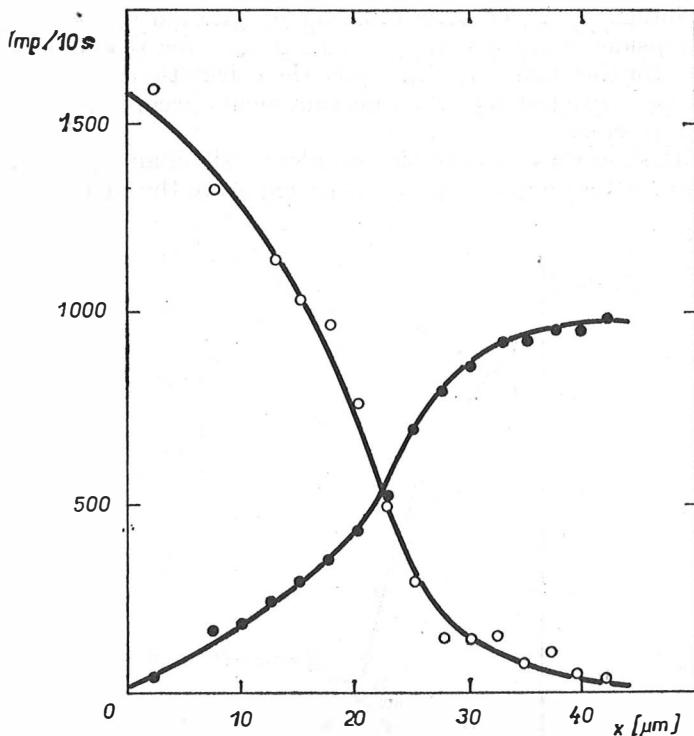


Fig. 4. Concentration profile of Ag and Na in glass surface layer (time 1 hr, 350 °C, AgNO_3 melt); ○ Ag, ● Na.

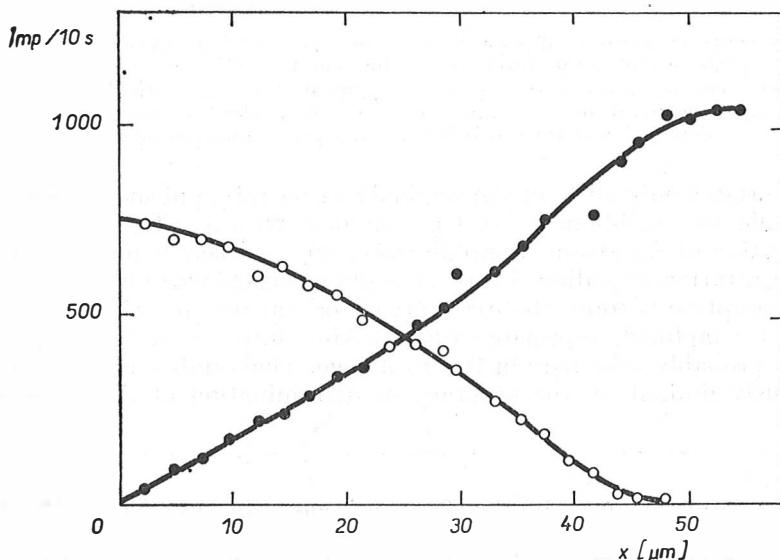


Fig. 5. Concentration profile of Ag and Na in glass surface layer (time 2.5 hrs, 350 °C, AgNO_3 melt); ○ Ag, ● Na.

of silver is higher in direct determination by gradual etching. These higher values are considered further on ($\rho_0 = 1.2 \text{ g cm}^{-3}$ for the time of 1 hr, $\rho_0 = 1.4 \text{ g cm}^{-3}$ for the time of 2.5 hrs) since the calculation of surface concentration on the basis of electron probe measurements according to an algorithm may involve an error.

Figs. 7 and 8 show the concentration profiles for silver and sodium expressed in moles per cm^3 . After one-hour temperature exposure the surface concentration

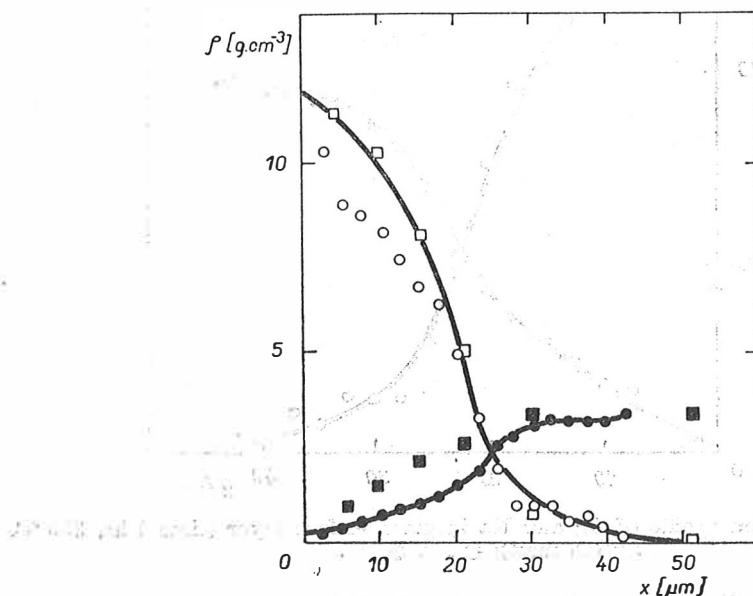
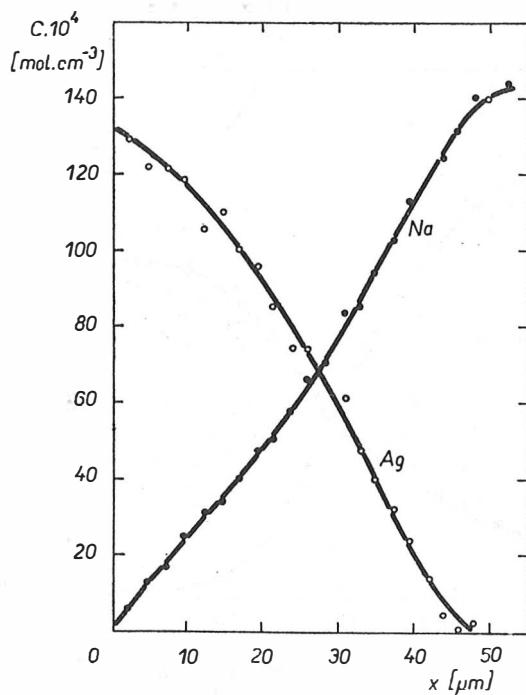
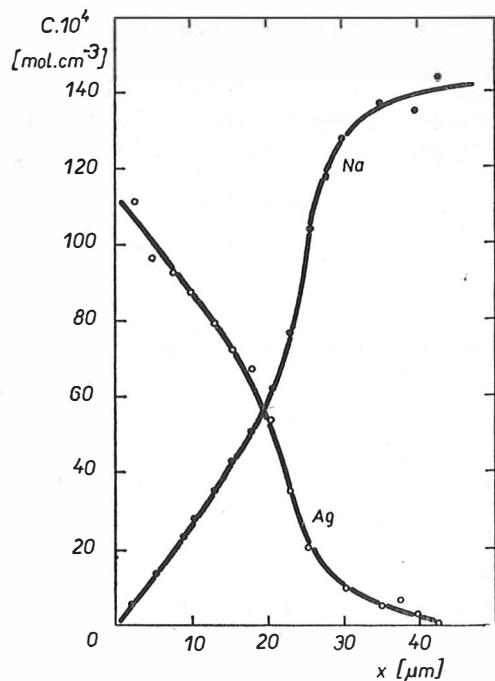


Fig. 6. Concentration profile of Ag and Na determined by electron probe measurements and by the gradual etching method (time 1 hr, 350 °C, AgNO_3 melt); □ Ag concentration determined by gradual etching, ■ Na concentration determined by gradual etching, ○ Ag concentration determined by electron probe measurements, ● Na concentration determined by electron probe measurements.

of silver attains only 80 % of the original concentration of sodium ions in the glass, while after 2.5-hour contact of the glass with AgNO_3 melt the surface concentration of Ag attains approximately 90 % of this value. In both cases the concentration of sodium ions in the glass surface was virtually zero. The usual assumption of equimolar exchange of sodium and silver ions thus appears somewhat simplified, especially when taking into account that potassium ions also probably take part in this exchange. The validity of this conclusion is obviously limited by the accuracy of determination of the concentration

Fig. 7. Concentration profile of Ag and Na in mole units (time 1 hr, 350 °C, AgNO_3 melt).

Fig. 8. Concentration profile of Ag and Na in mole units (time 2.5 hrs, 350 °C, AgNO_3 melt).



profiles. Especially in the proximity of phase boundary the concentration determination of the two ions involves a considerable error and may lead to unreliable conclusions.

From the point of view of technological practice it is important to determine the concentration of silver ions in the salt bath which would be satisfactory

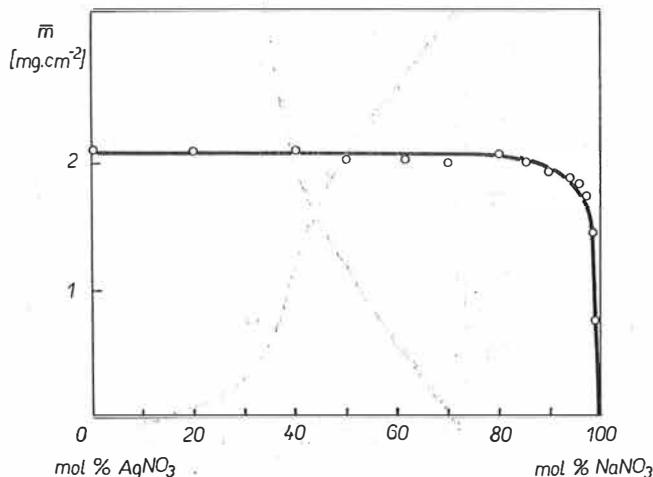


Fig. 9. Content of silver in glass vs. the concentration of AgNO_3 plus NaNO_3 mixture (time 1 hr, 350 °C).

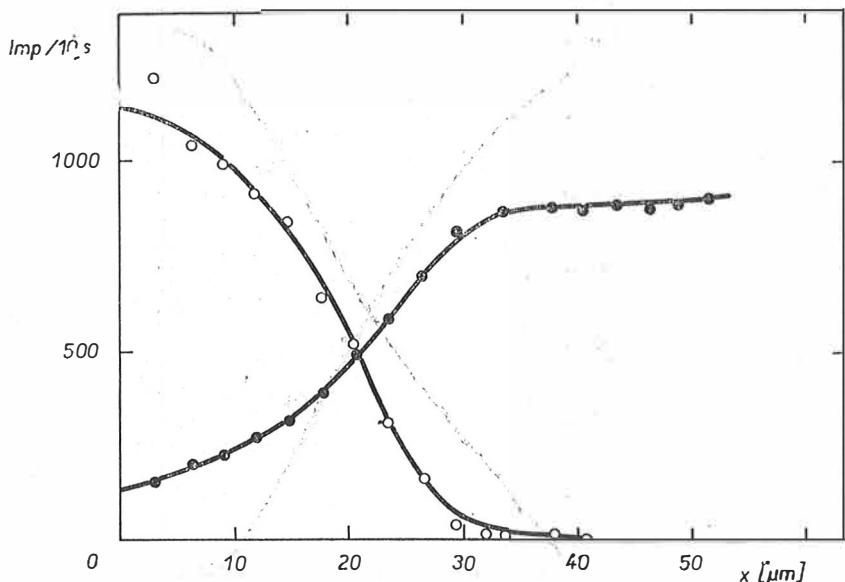


Fig. 10. Concentration profile of Ag and Na determined by electron probe measurements (time 1 hr, 350 °C, melt of 1.3 mole % AgNO_3 and 98.7 mole % NaNO_3).

for the preparation of diffusion layers identical as regards the content of silver and its distribution. As indicated by Fig. 9 the critical value of AgNO_3 concentration in mixture with NaNO_3 is approx. 20 mole %. The presence of NaNO_3 in the melt does not significantly affect the character of the concentration profile (cf. Fig. 10) although the surface concentration of silver is somewhat lower than in the preceding instances and Na concentration at the same sample point does not decrease to zero value. The equilibrium of the exchange reaction taking place at the phase boundary is therefore shifted to the detriment of silver ions.

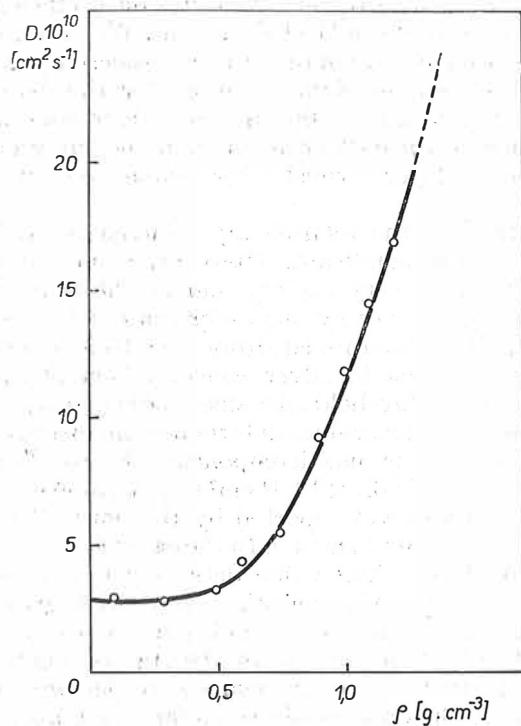


Fig. 11. The diffusion coefficient vs. the concentration of silver in glass.

The data mentioned so far allow to determine the value of parameters by means of which the concentration changes in the surface layer can be predicted. The linear relationship between silver content and the second root of time (cf. Fig. 2) is in agreement with the mathematical formula

$$m = 2\varrho_0 \sqrt{\frac{D t}{\pi}}, \quad (1)$$

where m is the Ag content per unit glass surface, ϱ_0 is the surface concentration of silver (density) in g cm^{-3} ; D is the mean value of the diffusion coefficient and t is the time of diffusion [4]. When considering the value $\varrho_0 = 1.4 \text{ g cm}^{-3}$

the mean diffusion coefficient $\bar{D} = 4.7 \times 10^{-10} \text{ cm}^2 \text{ s}^{-1}$. The concentration profiles of silver and sodium do not comply with the usual simple equation

$$\varrho = \varrho_0 \operatorname{erfc} \left(\frac{x}{2\sqrt{\bar{D}t}} \right), \quad (2)$$

where ϱ is concentration and x is the diffusion distance. The initial conditions and the boundary ones, under which this equation has been derived from the usual simple form of the IIInd Fick's law, have been very probably well complied with. The disagreement between experimental data and theory is therefore due to the assumption of a constant diffusion coefficient value which is included in the given form of the IIInd Fick's law. When neglecting the change in glass density with concentration of Ag (the respective data are not known) one may use the Boltzmann—Matan solution for the determination of the relationship between diffusion coefficient and silver concentration in glass. Fig. 11 shows this dependence determined from the silver concentration profile in the glass surface layer formed after 1-hour contact with the melt of pure AgNO_3 .

At lower concentrations the relationship between the diffusion coefficient and concentration is not significant. However, from the value of approx. 0.5 g cm^{-3} the D increases very rapidly and at the limit Ag concentration (1.4 g cm^{-3}) it is roughly higher by one order than at the zero concentration. The diffusion coefficient determined from the time dependence of silver content in glass corresponds to silver concentration of approx. 0.7 g cm^{-3} and belongs to the lower threshold of values indicated by the concentration profile. The diffusion coefficient values determined are in a very good agreement with the data published for multicomponent glasses. The mean \bar{D} value found by Ito [5] for 350°C is $6.7 \times 10^{-10} \text{ cm}^2 \text{ s}^{-1}$. A value of the diffusion coefficient identical in its order was reported by Doremus [6] from experimental data by Schulze $\bar{D} = 2.8 \times 10^{-10} \text{ cm}^2 \text{ s}^{-1}$. In the case of glass with a substantially lower Na_2O content (Pyrex) the value determined was lower by an order ($\bar{D} = 3.2 \times 10^{-11} \text{ cm}^2 \text{ s}^{-1}$) [7]. On the other hand with glass of a high Na_2O content (33.3 mole %) the diffusion coefficient values are in the range of 6×10^{-9} — $5.5 \times 10^{-8} \text{ cm}^2 \text{ s}^{-1}$ in relation to the Ag concentration in glass [8].

The diffusion coefficient values determined in the present study may be logically ranked among the data published so far which have been supplemented in particular with respect to the relationship between the diffusion coefficient and concentration. Knowledge of this relationship is indispensable for the preparation of diffusion layers with a defined concentration profile.

CONCLUSION

Interaction of glass with an AgNO_3 melt at 350°C brings about formation of surface layers in which the sodium ions have been approximately equimolarly replaced by silver ions. The content of silver in these layers increases linearly with the second root of time. The concentration changes affect a glass surface zone about 30 — $50 \mu\text{m}$ in thickness. The concentration of silver in glass surface attains at the most 90 % of the initial concentration of sodium ions in the glass. The content of silver and its distribution remains unchanged

when a mixture of AgNO_3 and NaNO_3 in concentrations of up to 80 mole % NaNO_3 has been used instead of the AgNO_3 melt.

Knowledge of the mean diffusion coefficient value and that of the surface concentration of silver in the given type of the surface concentration of silver in the given type of glass is indispensable when preparing layers with a defined Ag content. The mean value $D = 4.7 \times 10^{-10} \text{ cm}^2 \text{ s}^{-1}$ established from the time dependence of Ag content in glass corresponds approximately to the mean value of the concentration range of $0\text{--}1.4 \text{ g cm}^{-3}$ Ag. Within this range the diffusion coefficient is not constant as indicated by the analysis of concentration profiles, varying within the range of $3 \times 10^{-10}\text{--}1.7 \times 10^{-9} \text{ cm}^2 \text{ s}^{-1}$. The data determined within the framework of the present study are in a very satisfactory agreement with the published data on diffusion coefficients of silver in multi-component glasses.

Acknowledgement

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KONCENTRAČNÍ ZMĚNY V POVRCHOVÉ VRSTVĚ SKLA PŘI INTERAKCI S TAVENINOU AgNO_3

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Povrchové difúzní vrstvy s obsahem stříbra byly laboratorně připraveny ponorem vzorku měkkého technického skla ($68,8 \text{ SiO}_2$, $2,7 \text{ Al}_2\text{O}_3$, $4,0 \text{ MgO}$, $6,2 \text{ CaO}$, $0,8 \text{ K}_2\text{O}$, $17,3 \text{ Na}_2\text{O}$ — hmot. %) ve tvaru zkumavky do taveniny AgNO_3 nebo směsi AgNO_3 a NaNO_3 za teploty 350°C . V roztoce vzniklému odleptáváním povrchové vrstvy skla v HF byl metodou potenciometrické titrace stanoven obsah stříbra. Závislost obsahu Ag na druhé odmocnině z času je lineární (obr. 2). Pomocí elektronové mikroskopie byl určen koncentrační profil stříbrných a sodných iontů v povrchové vrstvě skla. Data o distribuci stříbra byla ověřena nezávislým stanovením metodou postupného leptání a určením obsahu stříbra v odleptaných vrstvách atomovou absorpcní spektrofotometrií. Koncentrační profily stříbra a sodíku (obr. 3—6) ukazují, že hloubka zóny, v níž dochází ke koncentračním změnám, činí $30\text{--}50 \mu\text{m}$ v závislosti na čase ($0,5\text{--}2,5 \text{ h}$). Koncentrace sodných iontů na povrchu skla klesá na nulovou hodnotu. Koncentrace stříbrných iontů dosahuje na stejném místě cca 80—90 % z původní hodnoty koncentrace iontů sodných (obr. 7—9).

Při použití směsi AgNO_3 s NaNO_3 se obsah stříbra a jeho rozdelení nemění, obsahuje-li směs dusičnanů více než 20 mol % AgNO_3 (obr. 9 a 10).

Data o obsahu a distribuci Ag v povrchové vrstvě skla byla použita pro výpočet difúzního koeficientu. Z časové závislosti obsahu Ag ve skle byla určena střední hodnota D pomocí vztahu (2). Pro hodnotu povrchové koncentrace $\varrho_0 = 1,4 \text{ g cm}^{-3}$ obdržíme $D = 4,7 \cdot 10^{-10} \text{ cm}^2 \text{ s}^{-1}$. Koncentrační profily Ag byly analyzovány použitím Boltzmanovy—Matanova metody a určena závislost difuzivity na koncentraci Ag. V koncentračním rozmezí $0\text{--}1,4 \text{ g cm}^{-3}$ se hodnota D mění v rozsahu $3 \cdot 10^{-11}$ až $1,7 \cdot 10^{-9} \text{ cm}^2 \text{ s}^{-1}$ (obr. 11). Střední hodnota D odpovídá přibližně střední hodnotě koncentračního rozmezí. Nalezené hodnoty difuzivity velmi dobře souhlasí s publikovanými údaji o difúzních koeficienzech stříbra v multikomponentních sklech.

Obr. 1. Aparatura pro tepelnou expozici vzorků; 1 — vzorek skla, 2 — termočlánek, 3 — kovová příručka, 4 — korundový kelímek, 5 — trubice z křemenného skla, 6 — tavenina, 7 — korundové víčko, 8 — korundová trubice, 9 — elektrická pec.

Obr. 2. Závislost obsahu stříbra ve skle na druhé odmocnině z času (350°C), tavenina AgNO_3 .

Obr. 3. Koncentrační profil Ag a Na v povrchové vrstvě skla (čas 0,5 h, 350°C , tavenina AgNO_3); ○ Ag, ● Na.

Obr. 4. Koncentrační profil Ag a Na v povrchové vrstvě skla (čas 1 h, 350°C , tavenina AgNO_2); ○ Ag, ● Na.

Obr. 5. Koncentrační profil Ag a Na v povrchové vrstvě skla (čas 2,5 h, 350°C , tavenina AgNO_3); ○ Ag, ● Na.

Obr. 6. Koncentrační profil Ag a Na určený na elektronové sondě a metodou postupného leptání (čas 1 h, 350°C , tavenina AgNO_3);

- koncentrace Ag určená postupným leptáním,
- koncentrace Na určená postupným leptáním,
- koncentrace Ag určená na elektronové sondě,
- koncentrace Na určená na elektronové sondě.

Obr. 7. Koncentrační profil Ag a Na v molových jednotkách (čas 1 h, 350°C , tavenina AgNO_3).

Obr. 8. Koncentrační profil Ag a Na v molových jednotkách (čas 2,5 h, 350°C , tavenina AgNO_3).

Obr. 9. Závislost obsahu stříbra ve skle na koncentraci směsi AgNO_3 a NaNO_3 (čas 1 h, 350°C).

Obr. 10. Koncentrační profil Ag a Na určený na elektronové sondě; (čas 1 h, 350°C ; tavenina 1,3 mol % AgNO_3 a 98,7 mol % NaNO_3).

Obr. 11. Závislost difúzního koeficientu na koncentraci stříbra ve skle.

КОНЦЕНТРАЦИОННЫЕ ИЗМЕНЕНИЯ В ПОВЕРХНОСТИНОМ СЛОЕ СТЕКЛА ПРИ ВЗАИМОДЕЙСТВИИ С РАСПЛАВОМ AgNO_3 И СО СМЕСЬЮ AgNO_3 И NaNO_3

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Лабораторным путем приготовлены поверхностные слои с содержанием серебра погружением пробы мягкого технического стекла ($68,8 \text{ SiO}_2$, $2,7 \text{ Al}_2\text{O}_3$, $4,0 \text{ MgO}$, $6,2 \text{ CaO}$, $0,8 \text{ K}_2\text{O}$, $17,3 \text{ Na}_2\text{O}$ % по весу) в виде химического стакана в расплав AgNO_3 или смеси AgNO_3 и NaNO_3 при температуре 350°C . В растворе, образовавшемся в результате травления поверхностного слоя стекла в НТ, определили содержание серебра методом потенциометрической титрации. Зависимость содержания Ag от второго корня времени линейна (рис. 2). С помощью электронного микрозонда определяли концентрационный профиль попов серебра и натрия в поверхностном слое стекла. Данные относительно распределения серебра проверяли независимым определением с помощью метода постепенного травления.

Содержания серебра в составе травленых слоев, было определено с помощью атомной абсорбционной спектрофотометрии. Концентрационные профили серебра и натрия показывают, что глубина зоны, в которой происходят концентрационные изменения, составляет в зависимости от времени (0,5—2,5 часа) 30—50 μm. Концентрация

ионов натрия на поверхности стекла понижается до нулевой величины. Концентрация ионов серебра на том же месте достигает приблизительно 80—90 % из исходной величины концентрации ионов натрия (рис. 7—9).

При применении смеси AgNO_3 с NaNO_3 содержание серебра и его распределение не изменяется тогда, когда смесь нитратов содержит больше чем 20 молей AgNO_3 (рис. 9 и 10).

Данные о содержании и распределении Ag в поверхностном слое использовали для расчета коэффициента диффузии. На основании зависимости содержания Ag в стекле определили среднюю величину \bar{D} с помощью уравнения (2). Для величины поверхностной концентрации $q_0 = 1,4 \text{ г. см}^{-3}$ получается $\bar{D} = 4,7 \cdot 10^{-10} \text{ см}^2 \text{ с}^{-1}$. Концентрационные профили Ag подвергали анализу с помощью метода Болцманн-Матана и определили зависимость диффузивности от концентрации Ag. В пределах концентрации 0—1,4 г cm^{-3} величина D изменяется от $3 \cdot 10^{-10}$ до $1,7 \cdot 10^{-9} \text{ см}^2 \text{ с}^{-1}$ (рис. 11). Средняя величина \bar{D} отвечает приблизительно средней величине пределов концентрации. Найденные величины диффузивностей находятся в хорошем согласии с опубликованными данными отсчитывая диффузионных коэффициентов серебра в многокомпонентных стеклах.

Рис. 1. Аппаратура для тепловой экспозиции проб; 1 — проба стекла, 2 — термометр, 3 — металлический фланец, 4 — корундовый тигель, 5 — трубка из кварцевого стекла, 6 — расплав, 7 — корундовый колпачок, 8 — корундовая трубка, 9 — электрическая печь.

Рис. 2. Зависимость содержания серебра в стекле от второго корня из времени (350°C , расплав AgNO_3).

Рис. 3. Концентрационный профиль Ag и Na в поверхностном слое стекла (время 0,5 ч., температура 350°C , расплав AgNO_3).

Рис. 4. Концентрационный профиль Ag и Na в поверхностном слое стекла (время 1 час, температура 350°C , расплав AgNO_3).

Рис. 5. Концентрационный профиль Ag и Na в поверхностном слое стекла (время 2,5 часа, температура 350°C , расплав AgNO_3).

Рис. 6. Концентрационный профиль Ag и Na, установленный на электронном зонде с помощью постепенного травления (время 1 ч., температура 350°C , расплав AgNO_3): \square — концентрация Ag, установленная постепенным травлением, \blacksquare — концентрация Na, установленная постепенным травлением, \circ — концентрация Ag, установленная на электронном зонде, \otimes — концентрация Na, установленная на электронном зонде.

Рис. 7. Концентрационный профиль Ag и Na в мольных единицах (время 1 ч., температура 350°C , расплав AgNO_3).

Рис. 8. Концентрационный профиль Ag и Na в мольных единицах (время 2,5 часа, температура 350°C , расплав AgNO_3).

Рис. 9. Зависимость содержания серебра в стекле от концентрации смеси AgNO_3 и NaNO_3 (время 1 ч., температура 350°C).

Рис. 10. Концентрационный профиль Ag и Na, установленный на электронном зонде (время 1 ч., температура 350°C , расплав 1,3 мол. % AgNO_3 и 98,7 мол. % NaNO_3).

Рис. 11. Зависимости коэффициента диффузии от концентрации серебра в стекле.