MATHEMATICAL MODEL OF Ag⁺—Na⁺ EXCHANGE IN THE SYSTEM FLOAT GLASS—AgNO₃ MELT

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Interaction of float glass with $AgNO_3$ melt involves a process whose rate is affected by the exchange of silver and sodium ions at the phase boundary, by diffusion of Ag^+ in the glass and by its reduction to Ag^0 by tin in the surface layer of the float glass. The experimental data on silver distribution in thin glass surface layer are best described by a mathematical model of diffusion concomitant by a chemical reaction. The rate of the chemical reaction is somewhat lower than would correspond to laminar diffusion. The equilibrium of the exidation-reduction reaction can be considered to be completely shifted in favour of the reduction of silver. The resulting concentration profile of silver is influenced by the relative content of various oxidation states of tin in the glass.

INTRODUCTION

Exchange of ions from glass for Ag^+ is sometimes used in the colouring of glasses. This exchange occurs whenever the glass is exposed to the effect of a melt, paste or atmosphere containing the given cation. The exchange cannot always be described by Fick's diffusion laws alone, because the diffusion can proceed together with further processes, e.g. reduction of Ag^+ to Ag° and the formation of nucleation centres. The reduction is caused by the presence of polyvalent elements in glass (Fe, Sb, As, Sn). These are mostly present in smaller amounts, either as residual refining agents or impurities from raw materials. An exception is the float glass, the bottom surface layer of which contains tin mostly in a lower oxidation state [1]. This is why the effect of reduction is more distinct in this glass.

The present study is based on the experimental data by Kočárek [2] which were obtained by submitting a float glass for 2, 4 and 8 hours to the effect of melt containing AgNO₃ at 350 °C. The concentration profiles of silver on both sides of the glass were measured by an electron microprobe. The present paper had the aim to describe the measuring results by a system of equations, the solution of which would give the concentration of silver as a function of depth in glass (x) and of the time of exchange (t).

$$w_{\mathrm{Ag}} = \bar{w}_{\mathrm{Ag}}(x, t).$$

KINETIC PROCESSES DURING THE EXCHANGE

The exchange can formally be described by the equation

$$Ag^{+} (melt) + Na^{+} (glass) = Ag^{+} (glass) + Na^{+} (melt).$$
(1)

The silver cations in glass may be further reduced according to the equation

$$\mathbf{mAg}^{+} + \mathbf{Sn}^{n+} = \mathbf{mAg}^{\circ} + \mathbf{Sn}^{(n+m)+}.$$
⁽²⁾

For the sake of simplicity, the only exchange considered is Ag^+ —Na⁺, because the mobility of Na⁺ is much higher than that of K⁺, which is the only further alkali

ion contained in distinct amounts in the float glass. For the same reason, just tin was regarded as a reduction agent, its content in the surface layer of the float glass is higher 10 to 100 times than that of the other reducing agents.

The entire process can be divided into the following five partial processes:

- 1. Diffusion of Ag⁺ in melt and that of Na⁺ in glass towards the phase boundary
- 2. The actual exchange at the boundary according to (1)
- 3. Diffusion of Ag⁺ in glass and that of Na⁺ in the melt from the boundary
- 4. Reduction of Ag⁺ according to [2]
- 5. Formation of the nucleation centres of Ag°.

The processes ad 3) and 4) are simultaneous, the others being sequential. The rate of the formation of the centres is therefore controlled by the slowest of the sequential processes. The mobilities of ions in the melt are higher by roughly two orders of magnitude than those in glass, and the mobility of Na⁺ exceeds that of Ag⁺ in the corresponding medium [3]. For this reason, process 1) and the second part of process 3) need not be considered.

Tin in glass will reduce just part of the silver that has diffused inside, while the rest can be reduced only after concluded exchange by the effect of reducing atmosphere. With respect to the colouring of glass it is therefore more important to know the total concentration distribution of silver after the exchange than the distribution of Ag°. For this reason process 5) has been neglected in further considerations, because silver is capable of diffusion in its ionic form only. The model should therefore include exchange at the boundary, diffusion of Ag⁺ in the glass and the reduction of Ag⁺.

MATHEMATICAL MODEL

On considering the Ag^+ —Na⁺ interdiffusion in glass just in the direction perpendicular to the phase boundary, the balance of Ag^+ weight provides the following equation:

$$\frac{\partial \varrho_{Ag^{+}}}{\partial t} = \frac{\partial}{\partial x} \left(D \frac{\partial \varrho_{Ag^{+}}}{\partial x} \right) + r_{Ag^{+}}, \tag{3}$$

which holds only under the assumption of neglecting the stresses arising during exchange in the glass surface, and the effect of the other components on the diffusion [4]. On describing the rate at which Ag^+ is produced by the chemical reaction by means of the equation known from the kinetics theory of chemical processes, equation (3) can be expressed in the form

$$\frac{\partial \varrho_{Ag^{*}}}{\partial t} = \frac{\partial}{\partial x} \left(D \cdot \frac{\partial \varrho_{Ag^{*}}}{\partial x} \right) - \frac{\partial \varrho_{Ag^{0}}}{\partial t},$$
$$\frac{\partial \varrho_{Ag^{0}}}{\partial t} = K(\varrho_{Ag^{\bullet}})^{f_{1}} (\varrho_{Sn^{n^{\bullet}}})^{f_{2}} - K\mathbf{1}(\varrho_{Ag^{0}})^{f_{3}} (\varrho_{Sn^{(m+n)^{+}}})^{f_{4}}.$$
(4)

The data on the basis of which the interdiffusion coefficient D was determined and which could be compared with the solution of the model are given in literature [2] in weight percent in the form $w_{Ag} = \bar{w}_{Ag}(x, t)$, $w_{Sn} = \bar{w}_{Sn}(x, t)$. The system [4] has therefore to be adjusted. On the assumption of a constant density of the glass one can write

$$\frac{\partial w_{Ag^{+}}}{\partial t} = \frac{\partial}{\partial x} \left(D \ \frac{\partial w_{Ag^{+}}}{\partial x} \right) - \frac{\partial w_{Ag^{0}}}{\partial t}$$
$$\frac{\partial w_{Ag^{0}}}{\partial t} = A \left(w_{Ag^{+}} \right)^{f_{1}} \left(w_{Sn} - \frac{M_{Sn}}{mM_{Ag}} w_{Ag^{0}} \right)^{f_{2}} - A I \left(w_{Ag^{0}} \right)^{(f_{3}+f_{4})} \left(\frac{M_{Sn}}{mM_{Ag}} \right)^{f_{4}} \qquad (5)$$
$$w_{Ag} = w_{Ag^{+}} + w_{Ag^{0}},$$

where

$$A = K \varrho^{(f_1 + f_2 - 1)}. \tag{6}$$

For Al holds an equation similar to that for A. The initial and the boundary conditions necessary for the resolving of system (5) are as follows:

$$\begin{array}{cccccccc} t = 0 & x = 0 & w_{Ag^{+}} = CP & w_{Ag^{0}} = 0 \\ & x > 0 & w_{Ag^{+}} = 0 & w_{Ag^{0}} = 0 \\ t > 0 & x = 0 & w_{Ag^{+}} = CP & w_{Ag^{0}} \ge 0 \\ & x > 0 & w_{Ag^{+}} \ge 0 & w_{Ag^{0}} \ge 0 \end{array}$$

No data on exchange reaction [1] were found, so that the surface concentration of Ag^+ (*CP*) has to be regarded as a function of time. No data on the reduction of silver with tin are likewise available. According to the mutual relationship between the diffusion rate of Ag^+ and its reduction, the following three cases can be distinguished:

a) The reduction is very slow. There will be virtually no reduction during the time the glass is exposed to the effect of the melt. The entire process can then be described by means of Fick's laws.

b) The rates of the two processes are comparable.

c) The reduction is very fast, taking place virtually in the front of the concentration profile of the diffusing silver. It is therefore a phenomenon similar to skin diffusion, which, in view of the planar symmetry of glass, should perhaps be better called laminar diffusion.

In the cases ad b) and c) the process has to be described by system (5) which can be resolved with values unknown from experimental measurement (A, A1, f1, f2, f3, f4, m, CP) in the function of parameters. The solution of the model of the individual values of these parameters was then compared with the experimental values [2]. The model did not include the diffusion of tin, since according to [5, 6, 7] the diffusion coefficient of all forms of tin in multicomponent glasses at 350 °C is lower by several orders of magnitude than the Ag^+ —Na⁺ interdiffusion coefficient.

NUMERICAL SOLUTION OF THE MODEL

The system (5) was solved by the numerical lattice method [8]. The values unknown from the experimental measurements were substituted from the following sets: $A \in \{10^{-5}, 10^{-4}, \ldots, 10^4\} h^{-1}, A/A1 \in \{10^{-3}, 10^{-2}, \ldots, 10^4\}, f1, f2, f3, f4 \in \{0, 1, 2, 3\}, CP \in \{16.5, 19.6, (-0.855t + 20.475), [exp (-0.0517t + 3.034)]\}$ wt. %. The first CP value is the mean of Ag⁺ concentrations measured on the glass surface after 2, 4, 8 hours, the second is the value after 2 hours, the time dependences were obtained by plotting the experimental values after 2, 4, 8 hours.

The tin was considered to be in oxidation state +2, i.e. m = n = 2. To determine the effect of the presence of the individual oxidation degrees of tin, several calculations

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were carried out on the assumption that just one of the degrees 0, +2, +4, is present over a certain range (Fig. 4). The diffusion coefficient, which depends on the concentration of Ag⁺, was determined by the Boltzmann—Matan procedure from the experimentally established concentration profile of silver on the non-tinned side of the glass, where the effect of reduction can be neglected. The relationship has the form

 $D_{350^{\circ}C} = 10.157 + 0.362 w_{Ag}^{2.2} \qquad [\mu m^2 h^{-1}].$ (7)

RESULTS OF NUMERICAL SOLUTION

The effect of A, f1, f2 on the solution of system (5)

The solution was found not to change if $A \leq 10^{-2-f_1}h^{-1}$. This can be explained so that the rate of reduction is then so low as to have practically no effect, and the solution corresponds to the model of plain diffusion. On the other hand, for high A $(\approx 10^4)$ h^{-1} , the reduction rate approaches that characterizing the laminar diffusion. A whole series of solutions obtained with various combinations of A, f1, f2 lie between these possibilities. A division is provided by the value $A = 10^{f_2 - f_1} h^{-1}$. A comparison of the solutions for the various values of A, f1, f2 is shown in Fig. 1. Over the interval $A \in \langle 0, 10^{f_2} - f^1 \rangle h^{-1}$, the gradient of the concentration profile of Ag increases in a depth over $15 \,\mu\text{m}$, which is understandable when considering that in greater distances from the boundary the reduction will decrease the driving force of diffusion, the concentration difference w_{Ag}^+ , the contrary being the case close to the boundary. It is interesting to note that this gradient again decreases with increasing A over the interval $A \in (10^{f_2-f_1}, \infty)$ h^{-1} . This can be explained so that at higher reduction rates the resulting changes have the time to equalize by diffusion during the time between the end of reduction and the end of the exchange. The differences in the distances over 15 µm from the boundary are of course relatively small, because in these glass layers the tin content and thus also that of Ag°, is already low.



Fig. 1. The effect of A, f1, f2 values on the solution of CP = 16.5 wt. %, tin as Sn^{2+} ; $1 - A = 10^{-2-f_1} h^{-1}$ (plain diffusion), $2 - A = 10^{f_2-f_1} h^{-1}$, $3 - A = 10^4 h^{-1}$, f1 = 0, f2 = 3(approaches the laminar diffusion), x – values established experimentally.

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The effect of CP on the solution of system (5)

As indicated by Fig. 2, the relationship $CP = \overline{CP}(t)$ has no significant effect on the solution. An almost identical course of the concentration profile with the assumed linear or exponential time dependence of CP, is given by a similar course of these curves over the time interval of $\langle 0, 2 \rangle h$, for which the model was solved. The absolute values of the solution are ifluenced rather more by the mean CP value than by its time dependence. Even at various absolute values, the shape of the concentration profile is not subject to any great changes.



Fig. 2. The effect of time dependence of concentration of Ag^+ at glass surface $(CP = \overline{CP}(t))$ on the solution; $A = 0.1 h^{-1}$, fI = f2 = 1, tin in the form of Sn^{2+} , I - CP = 16.5 wt. %, 2 - CP = 0.855 t + 20.475 wt. %, $3 - CP = \exp(-0.0517 t + 3.034) \text{ wt. }\%$, 4 - CP = 19.6 wt. %, x - values established experimentally.

The effect of reverse reaction (2) on the solution of (5)

This reaction, i.e. oxidation of Ag⁰ to Ag⁺, will become distinct only when the ratio $A/A1 \leq 10$. The solution for the individual values is shown in Fig. 3. The minute effect of the reverse reaction even at a small A/A1 ratio is due to the fact that during most of the time of reaction, $w_{Ag^+} \gg w_{Ag^0}$ or $w_{Ag^+} \gg w_{Sn^{(n+m)^+}}$. The inclusion of the reverse reaction into the model does not bring about distinctly different solutions, which could not be achieved on the assumption of shifting the equilibrium of reaction (2) completely to the right.

The effect of the presence of various oxidation states of tin on the solution of system (5)

This effect is considerable especially at the higher reduction rates, when virtually all tin is consumed, as indicated by Fig. 4. This is due to the fact that various amounts of Ag[°] correspond to tin oxidation states +2 and 0, as follows from the stoichiometry of the reduction. No reduction at all takes place when only Sn⁴⁺ is present. Fig. 4 also implies that mutual effects occur just between the closest points (x, w_{Ag}) . For example, solution in a region where Sn^{4+} is the only oxidation state present, is not affected by the oxidation state of tin in the neighbouring regions. This follows from the mathematical method chosen for the resolving.



Fig. 3. The effect of reverse reaction (8a) on the solution. CP = 16.5 wt. %; f1 = f2 = f3 = f4 = 1, $A = 1.0 h^{-1}$, tin in the form of Sn^{2+} , $1 - A1 = 10^3 h^{-1}$, $2 - A1 = 10^2 h^{-1}$, $3 - A1 = 10 h^{-1}$, $4 - A1 = 1 h^{-1}$, $5 - A1 = 0.1 h^{-1}$, x - values established experimentally.



Fig. 4. The effect of the presence of various oxidation states of tin on the solution, $CP = 16.5 \text{ wt. }\%, A = 10f^{2-f_1}h^{-1}$

1	Sn^{2+}	1 S	n⁴+ 1	Sn^{2+}
2	Sn ²⁺	1 S	n ⁴⁺ 1	Snº
3 3	Snº	, Sı	n ⁴⁺	Sn ²⁺
ō		6	13	x [µm]
x - value	ues esta	blishe	ed expe	rimentally

COMPARISON WITH EXPERIMENTAL DATA

This comparison is shown by Figs. 1 through 4. The solution closest to the experimental concentration profile is that for $A = 10^{f_2-f_1}h^{-1}$, $A/A1 \to \infty$, CP = 16.5 wt.%. However, even there is a considerably large deviation (Fig. 1). The reason of this was that the pulses of the electron microprobe were converted to concentration on the assumption of the same current passing through the sample and the standard. Different currents should be considered, because of the different composition of the sample and the standard. A comparison of the model solution with the converted experimental profile is shown in Fig. 5. The mean CP value for times 2, 4, 8 h was used in the calculation (*CP* 20.0 wt. %). The new dependence of the interdiffusion coefficient was as follows:

$$D_{350^{\circ}C} = 16.171 + 0.320(w_{Ag})^{2} \qquad [\mu m^{2} h^{-1}].$$
(8)

Following conversion, the model shows a quite satisfactory fit with the experimental data. The remaining deviations should be attributed to the simplifications introduced in the creation of the model. The most significant of these is the assumption of constant glass density during the exchange and neglection of the formation of a compressive stress on the glass surface.



Fig. 5. Comparison of some solutions with experimental values after conversion; CP = 20.0 wt. %, tin in the form of Sn^{2+} , $1 - A = 0.000 \ 1 \ h^{-1}$, f1 = 3, f2 = 1, 2 - A = 0. $1h^{-1}$, f1 = 1, f2 = 2, $3 - A = 0.01 \ h^{-1}$, f1 = 2, f2 = 2, or $A = 0.001 \ h^{-1}$, f1 = 3. f2 = 2, $4 - A = 10^{f_2 - f_1} \ h^{-1}$, x values established experimentally.

CONCLUSION

A mathematical model was proposed for the exchange of Ag^+ —Na⁺ ions in float glass, and its solution was verified for various rates of partial processes unknown from experimental measurements. The results can be summarized as follows:

1. A very rapid establishment of the exchange reaction and thus a constant concentration of Ag⁺ can be assumed at the glass melt boundary.

МАТЕМАТИЧЕСКАЯ МОДЕЛЬ ОБМЕНА Ag+-Na+ B CHCTE ME CTEKJIO FLOAT – PACIIJAB AgNO₃

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При окраске стекла Float оказывают влияние на кинетику процесса и на окончательный концентрационный профиль серебра в стекле следующие процессы: ионный обмен Ag+-Na на разделе фаз, диффузия Ag+ в стекле и его восстановление в Ago оловом, содержащимся в поверхности стекла.

Математическим описанием процесса является система (5) при следующих упрощающих предположениях: а) постоянная плотность стекла, б) влияние напряжения в поверхности стекла на распределение Ад+ пренебрежимо, в) диффузия протекает только в перпендикулярном направлении к разделу, г) на интердиффузию Ag+-Na+ а не оказывают влияние градиенты концентрации остальных компонентов стекла.

Так как из экспериментальных данных неизвестны величины A, A1, f1, f2, f3, f4, т, СР, выражающие скорость восстановления серебра (2) и равновесие поверхностного обменного взаимодействия (1), приходилось систему (5) решать, используя приводимые величины в функции параметров и ее отдельные решения сопоставляли с установленным экспериментальным путем профилем серебра (рис. 1—4). Лучше всего данным величинам отвечает решение модели, предполагающей быстрое установление равновесия обменной реакции (1), равновесие восстановления (2), сдвинутое совсем направо и скорость восстановления на несколько меньше, по сравнению с предполагаемым восстановлением, протекающим на фронте концентрационного профиля дифундирующего серебра (рис. 1, кривая 2). Отклонение от экспериментальных данных вызывается предположением постоянного тока, проходящего через образец и стандарт при измерении распределения Ад+ на электронном зонде. Если учитывается разность между приводимыми токами, то предложенная модель относительно хоропю отвечает процессу обмена (рис. 5, кривая 4).

Рис. 1. Влияние величин A, f1, f2 на решение, CP = 16,5 % по весу, олова в виде Sn^{2+} : $1 - A = 10^{-2} - 1h^{-1}$ (npocmas $\partial u \phi \phi \phi y \partial u s$);

$$2 - A = 10f^2 - f^{1h^{-1}},$$

 $3 - A = 10^{4}h^{-1}$, f = 0, f = 3 (близко диффузии, протекающей по слоям),

x — установленные экспериментальным путем величины.

Рис. 2. Влияние временной зависимости концентрации Ag+ на поверхности стекла $(CP = \overline{CP}(t))$ на решение:

- $A = 0, 1h^{-1}, f1 = f2 = 1, onobo e eude Sn^{2+}, 1 CP = 16,5 \%$ no eecy,

- $\begin{array}{l} 1 CF = 10.5 \ /_0 \ \text{no eccy}, \\ 2 CP = 0.855 \cdot t + 20.475 \ \% \ \text{no eccy}, \\ 3 CP = \varkappa c \ (-0.0517 \cdot t + 3.034) \ \% \ \text{no eccy}, \\ 4 CP = 19.6 \ \% \ \text{no eccy}, \end{array}$

х — установленные экспериментальным путем величины.

Рис. 3. Влияние обратной реакции (8а) на решение, CP = 16,5 % по весу, f1 = f2 = $= f3 = f4 = 1, A = 1,0h^{-1}$, onoso e sude Sn^{2+} .

- $1 A1 = 10^{3}h^{-1}$
- $2 A1 = 10^{2}h^{-1}$
- $\tilde{\boldsymbol{3}} \tilde{A1} = 10h^{-1},$
- $4-A1=1h^{-1},$
- $5 A1 = 0.1h^{-1}$.
- x установленные экспериментальным путем величины.
- Рис. 4. Влияние присутствия разных степеней окисления олово на решение, СР = 16,5% no secy, $A = 10f^{2-} f^{1}h^{-1}$

1	1	Sn ²⁺	i.	Sn⁴+	1	Sn ²⁺	
2	1	Sn ²⁺	I	Sn4+	I	Snº	
3	I	Sn⁰	I	Sn₄+	I	Sn²+	
	0		6		23		<i>х</i> [мм]

x — установленные экспериментальным путем величины.

Рис. 5. Сопоставление некоторых решений с экспериментальными величинами после пересчета:

CP = 20,0 % no secy, onose s sude Sn^{2+} , $1 - A = 0,0001h^{-1}$, f1 = 3, f2 = 2,

- $2 A = 0, 1h^{-1}, fI = 1, f2 = 2,$
- $3 A = 0,01h^{-1}, 1 = 2, 12 = 2$ unu $A = 0,001h^{-1}, 1 = 3, 12 = 2,$ $4 - A = 10^{t_2 - t_1} h^{-1}$

х — установленные экспериментальным путем величины.

BRODIE M. L., MYLOPOULOS J., SCHMIDT J. W.: ON CONCEPTUAL MODEL-LING (Koncepční modelování). Springer-Verlag, Berlin-Heidelberg-New York-Tokyo 1984. 510 str., cena 33.2 US dol.

Koncepční modelování se týká všech oblastí nauky o počítačích. Především jde o umělou inteligenci, datové soubory a programovací jyzaky. Kniha přináší první soubor výsledků výzkumných prací věnovaných koncepčnímu modelování. Je složena z devatenácti příspěvků, které byly předneseny na konferencích nebo byly publikovány v odborných časopisech.

V kapitole o perspektivách umělé inteligence jsou jednotlivé příspěvky věnovány popisu a programové specifikaci metodologického základu koncepčního modelování. Tato metodologie je v převážné míře užitečná pro informační systémy. Zajímavý v této kapitole je příspěvek C. Hewitta a P. de Jonga (Massachusetts Institute of Technology), který je věnován tzv. otevřeným systémům. Popis problémů spojených s otevřenými systémy úzce souvisí s koncepčním modelováním a tyto systémy je možno využívat při předvídání vývoje v přírodě.

Druhá část knihy je věnována perspektivám databází. Ve třech příspěvcích jsou popisovány pojmy, nástroje a techniky pro návrh a specifikaci chování databázových přenosů. Tyto pojmy, nástroje a techniky vyplývají ze souhrnu programovacích jazyků a databázových technologií.

Třetí část knihy je věnována perspektivám v programovacích jazycích. Značná část je věnována novým programovacím jazykům. Jde o nadmnožiny již používaných jazyků Pascal a Algol jako Pascal/R a PS-Algol. Velký prostor je zde věnován novému programovacímu jazyku ADA.

Poslední kapitola je věnována závěrečným poznámkám o již dříve zmíněných třech oblastech nauky o počítačích. Všechny příspěvky jsou zaměřeny na porovnání výsledků výzkumu se současným stavem a především je ukázán výhled do dalších let. Zřetelné je to zvláště u programovacích jazyků, kde nově vyvíjené nebo již vyvinuté jazyky dávají zcela jiné možnosti získání většího množství informací, co dosud používané jazyky (PL/1, Cobol aj.) nemohou ${f p}$ oskytnout. Tyto nové jazyky jsou vyvíjeny zvláště z důvodů možnosti předvídání stavů v lidské činnosti.

Kniha je doplněna velkým množstvím literárních odkazů (cca 300) a věcným rejstříkem. Z důvodu náročnosti popisované tématiky je nutné k jejímu studiu mít již dobré teoretické základy z nauky o počítačích, a proto je možné knihu doporučit okruhu specializovaných pracovníků v oblasti vývoje technického i programového zabezpečení počítačů.

Kasa

SCHOTT FORSCHUNGSBERICHTE 1981–1983 (Schott – výzkumné zprávy za léta 1981-1983). SCHOTT GLASWERKE, Mainz 1984. 416 str.

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Z předložených prací je zřejmý značný tematický rozsah výzkumu a vývoje, kterému se vědečtí a techničtí pracovníci firmy věnují, zejména v oblasti skel speciálních (11 prací) a skelně krystalických materiálů (3 práce).

Těžištěm výzkumné činnosti je v posledním desetiletí optimalizace určitých složení skel pro dané účely použití a nové možnosti zušlechťování speciálních skel. Např. vrstvy na skle nabízí řadu zajímavých možností použití (4 práce). Současně bylo třeba vyvinout odpovídající metody analýzy povrchu (3 práce), za účelem charakterizace jednotlivých vrstev a jejich kombinací.