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Stresses Produced in the BK7 Glass by the K^+Na^+ Ion Exchange: Real-Time Process Control Method

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Received: 25 February 2019; Accepted: 12 June 2019; Published: 21 June 2019



Featured Application: The paper describes a method allowing to control the chemical strengthening of glass by the ion exchange method. Process control is very simple; it comes down to current temperature measurements. The proposed method can find applications in industrial processes.

Abstract: The paper presents the results of tests on stresses produced by the $K^+ \leftrightarrow Na^+$ ion exchange method in BK7 glass. Diffusion ion exchange processes were carried out in glass plates with a surface area of a few cm^2 . The duration of these processes ranged from several hours to several hundred hours; process temperatures from 370 to 402 degree Celsius were used. The area of the glass in which the ion exchange took place shows refractive changes which are also accompanied by stresses. The planar waveguides produced in this way were tested by optical methods (for wavelength $\lambda = 677$ nm) and the refractive index profiles for the Transverse Electric (TE) and Transverse Magnetic (TM) polarization states were determined. On the basis of elasto-optic constants, the resulting stresses were determined. The temperature characteristics of diffusion coefficients of exchanged ions were also determined. Based on them a numerical simulation of real-time diffusion processes was possible, which allowed to predict the stresses arising in the glass. A good agreement between these predictions and the results of measurements was obtained.

Keywords: ion exchange in glass; diffusion; planar waveguides; stresses

1. Introduction

The mechanical properties of glasses are of great importance in practical applications. The possibility of glass strengthening (which is inherently a fragile material) plays an important role in the production of: display windows, in mobile personal electronic devices, cockpit windshields for aircrafts and vehicles, windows in architecture, panels for large displays, chemical equipment (pharmacy) and safeguard protections of all kinds [1]. In these cases, it is important to be able to modify the hardness of the glass surface. For this purpose, different technological methods are used [2]. In addition to the commonly used thermal method, the chemical ion exchange method is also utilized [3–7] at temperatures below the glass transition temperature. This method can be applied to finished glass products. The effects of the ion exchange process are the stresses created in the surface area of the glass. They have the character of compressive stresses and reach the maximum value (in the order of hundreds of MPa) at the glass surface [8]. The stresses fade away at depths from a few to tens of μm (depending on the depth of the ion exchange area) [9,10]. The resulting stresses are examined using various methods [2]. In addition to mechanical methods (determination of the modulus of rupture, impact testing), optical methods are also used [11,12]. Among them the waveguide method deserves more attention [13]. It is a non-destructive method, used for flat forms of glass, based on measurements of propagation constants of waveguide modes for TE and TM polarization [14]. Its advantage is the

possibility of determining the function of tension distribution from the surface into the glass. For its application, it is necessary to know the elasto-optic constants of a given type of glass.

The paper presents the use of this method for determining the function of stress distribution generated by $K^+ \leftrightarrow Na^+$ ion exchange in BK7 glass. A method of monitoring the occurring stresses (resulting from the ion exchange, based on the determination of diffusion profiles of dopant ions introduced into the glass) in the real time of the process [15] is also presented. The results obtained from the monitoring were compared with the results of optical measurements.

2. Theoretical Background

Changes in the refractive index in glasses, which are the effect of the resulting stresses, are determined by means of elasto-optic constants [16]:

$$C_1 = \frac{dn_{//}}{d\sigma} \text{ (Pa}^{-1}\text{)}, C_2 = \frac{dn_{\perp}}{d\sigma} \text{ (Pa}^{-1}\text{)} \quad (1)$$

In the above equations, $dn_{//}$ and $dn_{\perp}$ mean differentials of change in the refractive index for a wave with polarizations correspondingly parallel and perpendicular to the direction of stress σ . The elasto-optic constants thus defined can also be expressed by the following compounds [16]:

$$\begin{aligned} C_1 &= -\frac{n_0^3}{2E}(p_{11}-2\mu p_{12}) \\ C_2 &= -\frac{n_0^3}{2E}[-\mu p_{11} + (1-\mu)p_{12}], \end{aligned} \quad (2)$$

where:

n_0 —refractive index of the glass in the absence of stress,

p_{11}, p_{12} —elasto-optic coefficients,

E —Young's modulus,

μ —Poisson's ratio.

The knowledge of the elasto-optic coefficients p_{11} and p_{12} , the Young's modulus and the Poisson's ratio [16] allows, based on (2), to calculate the elasto-optic constants for the BK7 glass. These values for the wavelength $\lambda = 677 \text{ nm}$ are [17]:

$$C_1 = -0.5 \times 10^{-6} \text{ (MPa}^{-1}\text{)}, C_2 = -3.3 \times 10^{-6} \text{ (MPa}^{-1}\text{)} \quad (3)$$

The processes of ion exchange in glass with the use of a liquid source of admixture ions, among which the most widely used are nitrates, are carried out at temperatures much lower than the transition temperature of glasses T_g . For the borosilicate glass BK-7, this temperature $T_g = 557^\circ\text{C}$ [16] is much higher than the temperature $T_{diff} \sim 400^\circ\text{C}$ of the implementation of the $K^+ \leftrightarrow Na^+$ ion exchange processes with the use of liquid potassium nitrate KNO_3 as the source of K^+ ions. In such cases, changing the refractive index of the glass in its surface area where the ion exchange occurs is due to not only the difference of their electric polarizability, but also as the result of the elasto-optic phenomenon generated by mechanical stresses occurring in this area [13]. These stresses are the result of changes in the volume of glass in the doping area, which results from the difference of ionic radii of exchanged components, as well as the difference in thermal expansion between the doped region and the rest of the glass.

The geometry of these stresses is shown in Figure 1. The presence of these stresses in the waveguide layer of the glass is revealed during the propagation of the electromagnetic wave in it. Then there is a difference between the constant propagation of the modes of the same order for a monochromatic

wave, depending on the state of its polarization: TE or TM. The refractive index profiles corresponding to these polarization states are described in the following equations [13]:

$$\begin{aligned} n_{TM}(x) &= n_0(x) + C_1\sigma_{xx}(x) + C_2[\sigma_{yx}(x) + \sigma_{zx}(x)] \\ n_{TE}(x) &= n_0(x) + C_1\sigma_{yx}(x) + C_2[\sigma_{xx}(x) + \sigma_{zx}(x)], \end{aligned} \quad (4)$$

where: $n_{TM}(x)$, $n_{TE}(x)$ —refractive index profiles for TM and TE polarizations, $n_0(x)$ —refractive index profile of a waveguide in the absence of stresses.

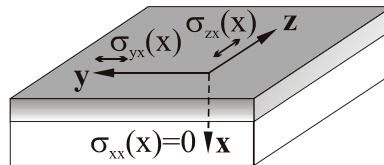


Figure 1. Stress directions in a planar waveguide.

After taking into account the assumptions: $\sigma_{xx}(x) = 0$ and $\sigma_{yx}(x) = \sigma_{zx}(x) = \sigma(x)$, the Equation (4) simplify to the form:

$$\begin{aligned} n_{TM}(x) &= n_0(x) + 2C_2\sigma(x) \\ n_{TE}(x) &= n_0(x) + (C_1 + C_2)\sigma(x) \end{aligned} \quad (5)$$

They allow to determine the value of stresses created in the doping area of the glass. From the Equation (5) results the following:

$$\sigma(x) = \frac{n_{TM}(x) - n_{TE}(x)}{C_2 - C_1} \quad (6)$$

The refractive index profiles of a waveguide for TE and TM polarization and the refractive index profile in the absence of stresses can be represented in the form [14]:

$$\begin{aligned} n_{TE}(x) &= n_b + \Delta n_{TE}u(x) \\ n_{TM}(x) &= n_b + \Delta n_{TM}u(x) \\ n_0(x) &= n_b + \Delta n_0u(x) \end{aligned} \quad (7)$$

In the above equations, n_b means the refractive index of the glass (without ion exchange), Δn_i is the increase in the refractive index at the glass surface (for $i = TE, TM, 0$), $u(x)$ is a function that describes the normalized concentration of the admixture ions introduced into the glass [14]. These dependencies are shown in Figure 2.

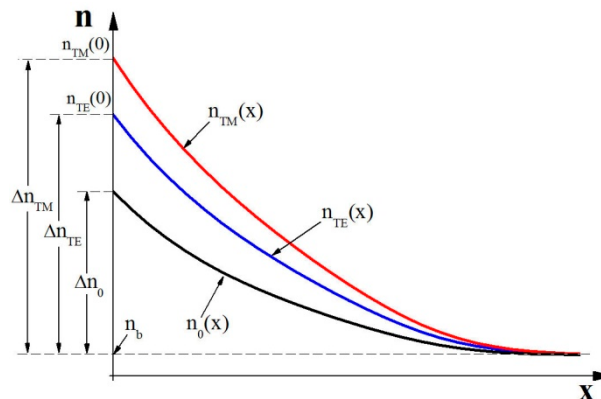


Figure 2. Refractive index profiles in a planar waveguide showing birefringence.

The refractive index profile $n_0(x)$, in the absence of stresses, can be expressed on the basis of (5) in two ways:

$$\begin{aligned} n_0(x) &= n_{TM}(x) - \frac{2C_2}{C_2 - C_1} [n_{TM}(x) - n_{TE}(x)] \\ n_0(x) &= n_{TE}(x) - \frac{C_2 + C_1}{C_2 - C_1} [n_{TM}(x) - n_{TE}(x)] \end{aligned} \quad (8)$$

Taking into account dependencies (7) in the Equation (6) we get:

$$\sigma(x) = \frac{n_{TM}(0) - n_{TE}(0)}{C_2 - C_1} \times u(x) = \frac{n_{TM}(0) - n_{TE}(0)}{C_2 - C_1} \times \frac{n_0(x) - n_b}{\Delta n_0} = \sigma(0) \times \frac{n_0(x) - n_b}{\Delta n_0} \quad (9)$$

The obtained dependence (9) links the function of stresses $\sigma(x)$ with the refractive index profile $n_0(x)$. This compound plays an important role in modeling stresses. The idea of modeling the refractive index profiles in the real time of the ion exchange process [10,14] can be applied here in relation to $\sigma(x)$ function, which is shown in Figure 3. The $\sigma(x)$ function calculations included stress relaxation at the glass surface according to the equation [6,18]:

$$\sigma(0, t) = \sigma(0, 0) \exp[-(t/\tau)^\beta] \quad (10)$$

where: $\sigma(0, 0)$ is the surface compressive stress at the beginning of the diffusion process, τ is the relaxation time, β is the stretching exponent.

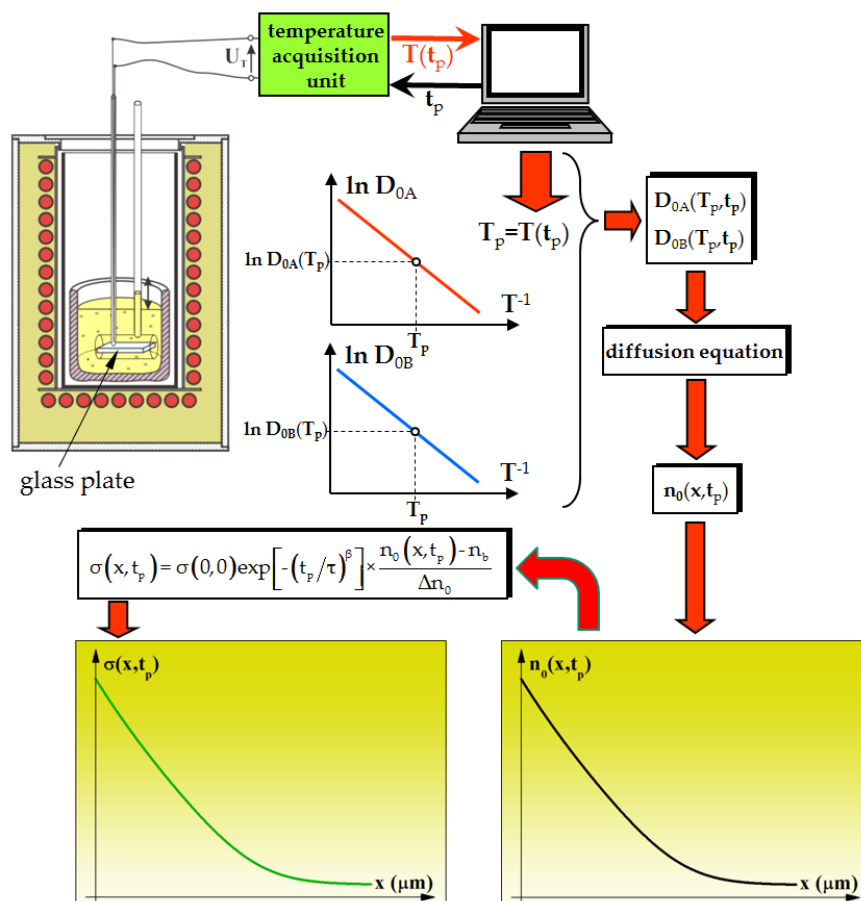


Figure 3. The principle of monitoring stresses generated in glass in real time based on temperature measurements.

According to the scheme shown in Figure 3, the diffusion equation is integrated here in the time domain with the Δt time step [14,15]. Solving this equation is carried out in parallel with the

implementation of the diffusion process. The temperature in the crucible is measured at specific moments of time t_p , which are the total multiplicity of the time step Δt . The diffusion coefficients occurring in this equation are calculated in moments of time t_p based on the knowledge of their temperature dependencies. The refractive index profile $n_0(x, t_p)$ without stresses is calculated here at discrete time points t_p . On its basis and at the same time points, the function $\sigma(x, t_p)$ given by the following equation is calculated:

$$\sigma(x, t_p) = \sigma(0, 0) \exp\left[-(t_p/\tau)^\beta\right] \times \frac{n_0(x, t_p) - n_b}{\Delta n_0} \quad (11)$$

3. Experiment

The method mentioned above was used to monitor the function describing the stresses produced by $K^+ \leftrightarrow Na^+$ ion exchange in BK7 glass (from Schott AG, Mainz, Germany). Flat glass plates with dimensions of $12 \times 30 \times 1.5$ mm were used. The source of K^+ ions was KNO_3 sodium nitrate (pure for analysis). The volume of salt melted in the ceramic crucible was ~ 250 cm³. The glass plates were introduced into the crucible in a special holder made of fused quartz [14]. Before immersion in the molten salt they were gradually heated and after removal they were slowly cooled down and washed in deionized water. The measurements of propagation constants of waveguide modes were made using the m-line method by measuring synchronous angles [14]. These measurements were carried out using a goniometer with an angular resolution of $1.8''$. The light source was a laser diode ($\lambda = 677$ nm). A prismatic coupler made of PSK-3 glass (from Schott AG, Mainz, Germany) was used (refractive index $n_{677} = 1.5491$, breaking angle $\delta = 70.0963^\circ$). The uncertainties of the determined effective refractive indexes of the waveguide modes were of the order of $\sim 10^{-4}$ [15]. The refractive index profiles were calculated using a procedure based on the mode characteristic equation [14,19]. In this case, the number of waveguide modes determines the number of points of the refractive index profile. The function describing the $n_{TE}(x)$ or $n_{TM}(x)$ refractive index profiles was obtained by fitting the solution of the diffusion equation to the points of the profile [14]. Then, on the basis of Equation (8), $n_0(x)$ was calculated. An example of such calculations is shown in Figure 4.

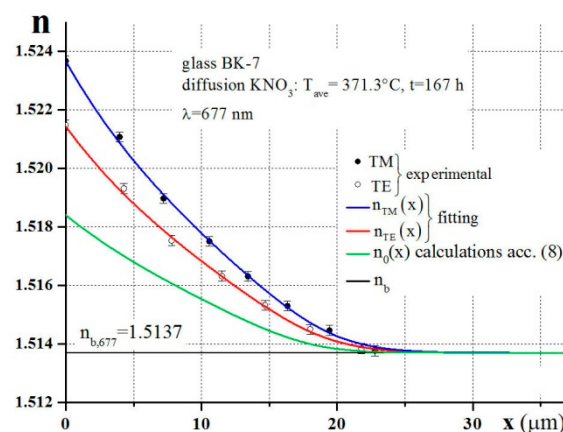


Figure 4. An example of profile determination $n_0(x)$ according to (8).

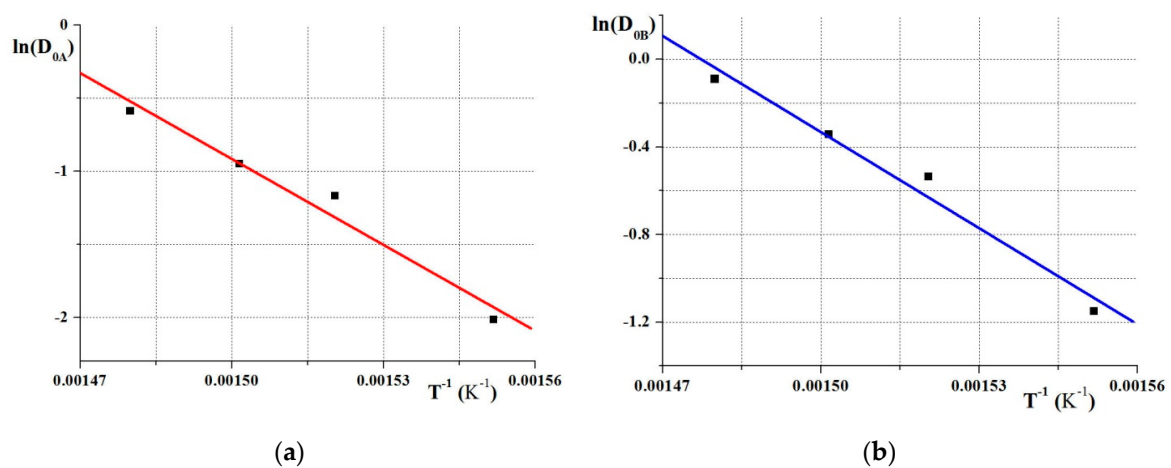
3.1. Determining the Dependence of Diffusion Coefficients on Temperature

The above-mentioned principle of monitoring the $\sigma(x, t)$ function requires knowledge of the temperature dependencies of the diffusion coefficients of exchanged ions. These coefficients are used to determine the $n_0(x)$ refractive index profile in the waveguide in the absence of stresses. Four ion exchange processes were carried out. The data of these processes are presented in Table 1.

Table 1. Parameters of ion exchange processes for determination of temperature dependencies of diffusion coefficients.

Process Number	Average Temperature (°C)	Time (h)
1	371.3	167
2	384.6	118
3	392.9	116
4	402.6	73

The refractive index profiles $n_0(x)$ for four temperature values were then calculated using the method described above. By adjusting the solution of the diffusion equation to each of the designated in that way $n_0(x)$ profiles, the temperature dependencies of diffusion coefficients of the exchanged ions were determined [14]. Using the linearized form of the Arrhenius equation, the values of coefficients describing $D_{0A}(T)$ and $D_{0B}(T)$ temperature dependencies [15] were determined. An illustration of these dependencies is shown in Figure 5.

**Figure 5.** The temperature dependencies of diffusion coefficients (according to the linearized form of the Arrhenius equation) for $K^+ \leftrightarrow Na^+$ ion exchange in BK7 glass. (a) D_{0A} (K^+ ions), (b) D_{0B} (Na^+ ions).

The determined coefficients were collected in Table 2. The symbol Δn_{0ave} denotes here the average value Δn_0 of the four determined refractive index profiles $n_0(x)$.

Table 2. Diffusion coefficients.

$\Delta Q_A/R$ (K)	$\ln(D_{0A}^*)$	$\Delta Q_B/R$ (K)	$\ln(D_{0B}^*)$	$n_{b,677 \text{ nm}}$	Δn_{0ave}
$-1.9592 \cdot 10^4$	28.473	$-1.4627 \cdot 10^4$	21.609	1.5137	0.0047

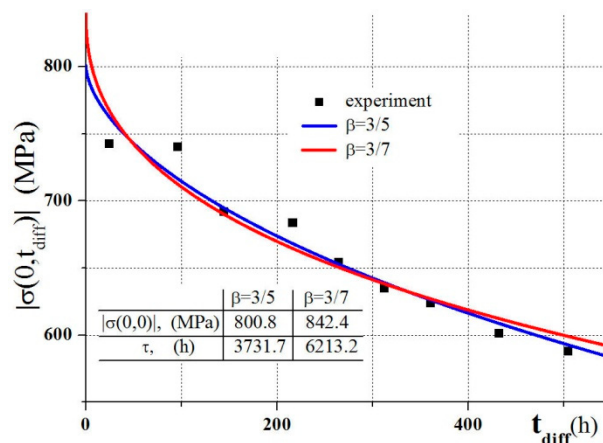
3.2. The Relationship between the Stress Relaxation and the Diffusion Duration

In order to determine the dependencies of stress relaxation at the surface of the glass on the duration of the diffusion process, nine long-term diffusion processes were carried out with the duration of: from 24 to 504 h. With a long process duration, the contents of the crucible were repeatedly replenished by providing a portion of potassium nitrate. The purpose of this procedure was to ensure a constant efficiency of the source of the admixture ions. The average temperatures of these processes were close to 400 °C. The parameters of these processes are summarized in Table 3.

Table 3. The parameters of long-term $K^+ \leftrightarrow Na^+$ ion exchange processes in BK7 glass.

Process Number	Average Temperature (°C)	Time (h)	$\sigma(0, t_{diff})$ (MPa)
1	400.2	24	742.8
2	399.5	96	740.4
3	399.4	144	692.1
4	399.1	216	683.9
5	399.2	264	654.3
6	399.4	312	635.0
7	399.7	360	624.3
8	399.7	432	601.4
9	399.6	504	588.2

The measurements of effective refractive indices of the modes for the produced waveguides were carried out (TE and TM polarization). Using the procedure based on the mode characteristic equation, the values of $n_{TM}(0)$ and $n_{TE}(0)$ [19] were determined. By substituting the values of the elasto-optic constants (3) to the Equation (6), the stresses $\sigma(0, t_{diff})$ generated at the surface of the glass were calculated (Table 3). Figure 6 shows the calculated values of these stresses as a function of the diffusion duration. The adjusted curves (Equation (10)) correspond to the model of short-range stress relaxation ($\beta = 3/5$) and structural relaxation ($\beta = 3/7$) [20].

**Figure 6.** The exponents matched for the Equation (10) and the determined parameters for the model of short-range stress relaxation ($\beta = 3/5$) and structural relaxation ($\beta = 3/7$).

The parameters determined in this way allow to numerically model the course of the function $\sigma(x, t)$ using the Equation (11). The next chapter presents the results of such modelings made in the real time of the ion exchange process based on the measurement of the current process temperature.

4. Numerical Simulations and Measurements

The principle of monitoring stresses in the real time of diffusion processes (according to Figure 3) has been verified. This verification consisted in comparing the stress values obtained from measurements of glass samples after diffusion processes with the results of a real-time simulation. The measurements were made using the m-line method mentioned above. The effective refractive indexes for TM and TE polarization were determined and the stress values were specified based on (6). The calculation method is shown in Figure 7.

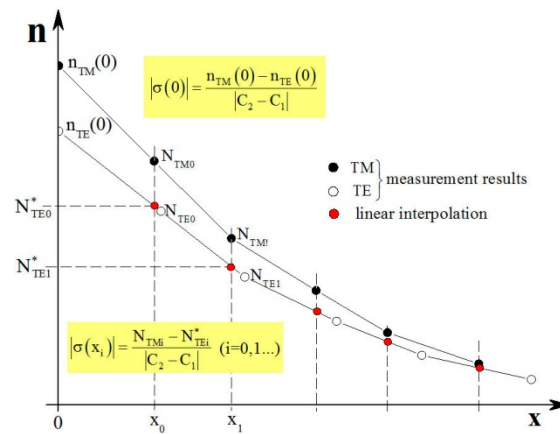


Figure 7. The method of calculating stresses based on the measurements of effective refractive indices of the modes.

The N_{TEi}^* values (visible in Figure 7) were calculated using linear interpolation. For each sample, a set of values $\{x_i, |\sigma(x_i)|\}$ for $i = 0..m$ was obtained, where m is the maximum TM mod row adopted for the calculation. Such calculations are possible for processes that result in multimode waveguides. Figure 8a shows an example of comparison of the measurement results with the final course of the $\sigma(x)$ function, simulated during the process of diffusion doping of BK7 glass with K^+ ions.

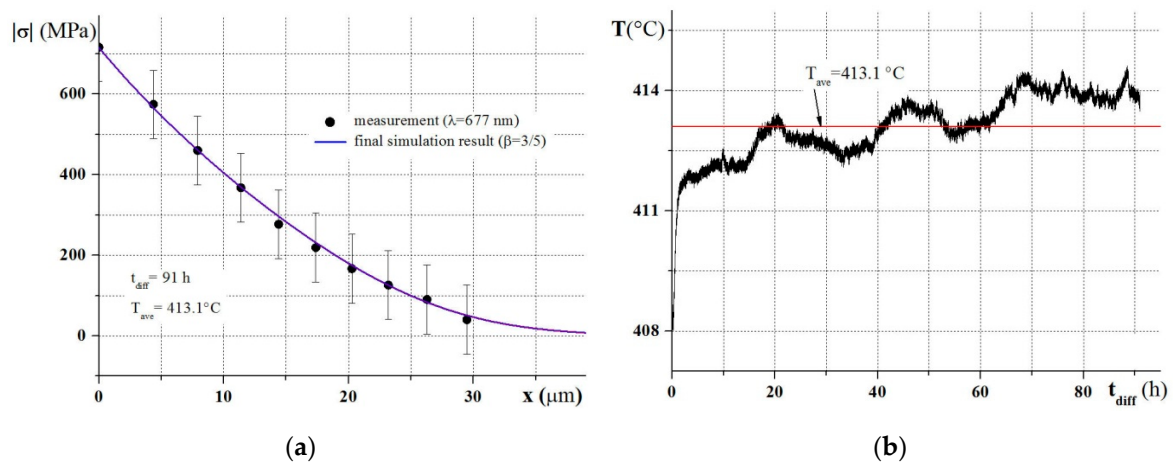


Figure 8. A comparison of the measurement results ($\lambda = 677$ nm) with simulation calculation of the $\sigma(x)$ function (a). The time course of the process temperature (b).

The duration of the diffusion process was 91 h and the average temperature of the process was 413.1 °C. The produced waveguide supported 10 TM and 10 TE modes. The calculations shown in Figure 8a were made in this case for 9 TM modes. Figure 8b shows the recorded course of the process temperature. In the simulation process in the Equation (11) the $\beta = 3/5$ exponent was assumed.

As can be seen in Figure 8a, the final result of the numerical simulation is in a very good agreement with the measurement results.

Figure 9a shows similar comparisons for three diffusion processes carried out in the following conditions: (1) $t_{diff} = 30$ h; $T_{ave} = 400.3$ °C, (2) $t_{diff} = 72$ h; $T_{ave} = 399.8$ °C and (3) $t_{diff} = 240$ h; $T_{ave} = 399.0$ °C. The uncertainties $\Delta\sigma$ calculated on the basis of measurements were also marked there. A good compliance between the numerical simulations and the results of the final measurements occurs here as well. Also, in these simulations, the exponent $\beta = 3/5$ was assumed in the Equation (11).

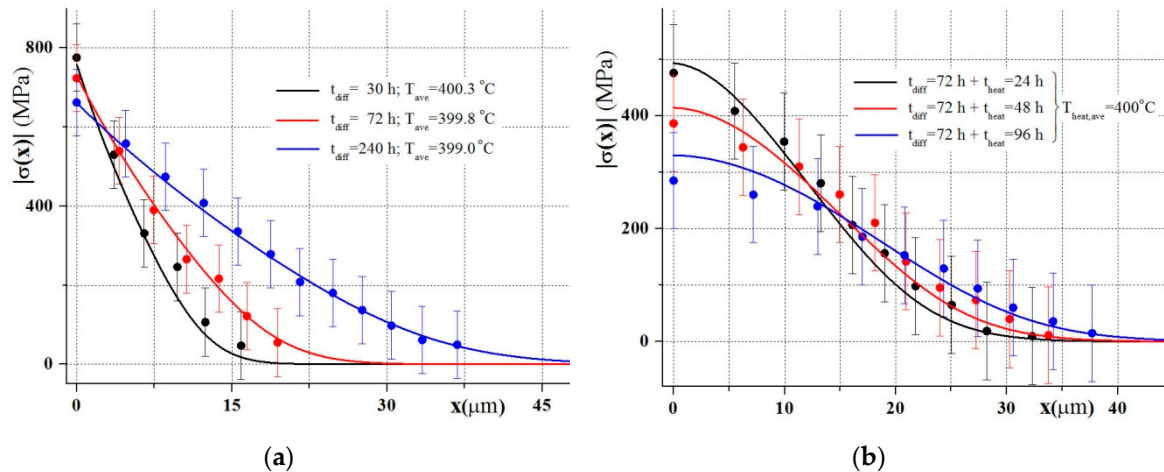


Figure 9. A comparison of simulation results of the $\sigma(x)$ function and the measurements ($\lambda = 677$ nm) of (a) diffusion processes, (b) heating processes.

Figure 9b presents the results of the simulation of the $\sigma(x)$ function in the case of heating processes. These processes were carried out for a sample of glass previously subjected to a diffusion process with a duration $t_{diff} = 72$ h. The $\sigma(x)$ function for this sample is shown in Figure 9a. After the diffusion process the sample was cut into three pieces and each of them was subjected to heating where the average value of the temperature was 400 °C. As can be seen from the results presented in Figure 9b, the simulation's compliance with the measurement results is slightly worse here than in the case of diffusion processes. This applies especially to the initial fragments of the course of the $\sigma(x)$ function. As in the previous simulations, the $\beta = 3/5$ exponent from Equation (11) was assumed here.

5. Discussion

The paper presents a method of real-time monitoring of stresses induced in glass during ion exchange processes. The presented results refer to the BK7 glass and $K^+ \leftrightarrow Na^+$ ion exchange. The proposed method is non-destructive and is based on the knowledge of the elasto-optic constants of the glass. It is also necessary to know the temperature dependence of the kinetics of the ion exchange in glass. Additionally, the knowledge of the function describing the time relaxation of stresses at the glass surface is required.

In all numerical calculations simulating the shaping of the $\sigma(x)$ function, it was assumed that the stress relaxation time τ (Equations (10) and (11)) is isothermal. The justification is the actual temperature of the processes for which the simulations were used according to (11). In all cases, they do not differ by more than 3 °C from the temperature of 400 °C. A large temperature span of processes (over 30 °C) occurs only in the case of determining the temperature dependencies of diffusion coefficients (see data in Table 1).

The results of the determination of the $\sigma(x)$ function were compared in this paper with the results of measurements made using the optical method. The basis of such proceedings is the knowledge of the elasto-optic constants of a given glass. The measurement verification required performing long (with a duration of over 24 h) diffusion processes. It resulted from the necessity to determine the refractive index profile using the waveguide method (multimode waveguides), which allows to determine the values of effective refractive indexes with an uncertainty of $\Delta N \sim 10^{-4}$. This translates into the uncertainties $\Delta\sigma$ of several dozens of MPa. Using the waveguide method based on the mode characteristic equation, it is not possible to determine refractive index profiles for single-mode waveguides. Fidelity to the reconstruction of the refractive index profile using this procedure increases with the number of modes conducted through the waveguide [21]. For this reason, the verification of stresses arising as a result of short diffusion processes is not presented here. This is however not an obstacle to numerical simulation of such processes according to (11).

The method described in this paper has been used to simulate the one-dimensional stresses and is based on simulating the refractive index profile of a planar structure. It is also possible to simulate (in the real time of the technological process) the two-dimensional refractive index profiles $n(x,y)$ and, therefore, to simulate a two-dimensional form of stress function $\sigma(x,y)$.

6. Conclusions

The paper describes a method allowing to control the chemical strengthening of glass by the ion exchange method. The conducted research concerns the BK7 glass and $K^+ \leftrightarrow Na^+$ ion exchange. The presented method can, however, be applied to a different type of glass and other admixture ions. Process control is very simple; it comes down to current temperature measurements. The proposed method can find applications in industrial processes.

Funding: This research received no external funding.

Conflicts of Interest: The author declare no conflict of interest.

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