

EXPERIMENTAL AND THEORETICAL INVESTIGATION OF THE ION-MASKING

EFFECT IN PLANAR OPTICAL WAVEGUIDES FABRICATED BY THE

DOUBLE-ION EXCHANGE TECHNIQUE IN GLASS SUBSTRATES

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ABSTRACT:

Previous works have shown that, using two consecutive ion-exchange processes with two different ions, it is possible to fabricate, in the same planar optical waveguide, two different regions having different effective refractive indices. In this paper we present a quantitative experimental and theoretical study of the double-ion exchange process. It is shown that an ion-exchange process with one species of ions halts a subsequent exchange process with another different ion. The refractive index profile of the resulting waveguide is derived from the measured values of the propagation constants of the guided modes using an inverse scattering method. We demonstrated that a third-order polynomial provides a good enough description of the measured refractive index profile; the different parameters involved in the double-ion exchange process can then be deduced by comparison with an infinite power series solution.

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**ABSTRACT:** Previous works have shown that, using two consecutive ion-exchange processes with two different ions, it is possible to fabricate, in the same planar optical waveguide, two different regions having different effective refractive indices. In this paper we present a quantitative experimental and theoretical study of the double-ion exchange process. It is shown that an ion-exchange process with one species of ions halts a subsequent exchange process with another different ion. The refractive index profile of the resulting waveguide is derived from the measured values of the propagation constants of the guided modes using an inverse scattering method. We demonstrated that a third-order polynomial provides a good enough description of the measured refractive index profile; the different parameters involved in the double-ion exchange process can then be deduced by comparison with an infinite power series solution.

Introduction:

Glass waveguides are suitable candidates for a class of passive integrated optical components such as star and access couplers, beam splitters, power combiners, ring resonators etc... The use of glass as

the building block for such components is attractive for reasons such as low loss, compatibility with commercial fibre interfacing, and potential low cost. The ion-exchange method is the most powerful technique because of its simplicity and flexibility in choosing the numerical aperture and dimensions of the waveguides. Many investigators have reported results on the ion-exchange properties of glass in binary molten salt media<sup>(1-4)</sup>.

Chartier et. al.<sup>(5)</sup> have experimented with a double-ion exchange in the same glass substrate, firstly with potassium at 390°C, and subsequently with silver at 225°C (using a pure melt of AgNO<sub>3</sub>) or at 215°C (using dilute melt of 5% molar AgNO<sub>3</sub> in NaNO<sub>3</sub>). They found that a shallow exchange of potassium into a soda-lime glass slows the subsequent exchange of silver, by a significant amount in the dilute melt, and almost to a standstill in pure silver nitrate. They claimed that this effect may be due to the reduced self-diffusion coefficient of potassium at the lower silver exchange temperatures. The value of the effective diffusion coefficient  $D_e$  of the K<sup>+</sup>/Na<sup>+</sup> exchange is two to three orders of magnitude lower than the corresponding value for the Ag<sup>+</sup>/Na<sup>+</sup> exchange. As a small self-diffusion coefficient will dominate any exchange process<sup>(1)</sup>, it is presumably the slow diffusion of potassium which is regulating the silver exchange into a previously potassium-exchanged substrate, the potassium exchanged areas at the surface of the waveguide act as a mask for a next silver exchange.

In this paper we present a quantitative experimental verification of this masking effect in the case of double-ion exchanged waveguides.



We also demonstrate that the refractive index distribution resulting from such a process can be described by a third-order polynomial rather than by a second-order one<sup>[4]</sup>. A comparison between the infinite power series solution of the diffusion equation and the third-order polynomial enables us to calculate the depth parameter and the effective diffusion coefficient involved in the double-ion exchange process.

#### 1- Experimental verification of the ion-masking effect:

We performed two sets of experiments. In the first one, a microscope glass slide was immersed in a molten bath of  $\text{KNO}_3$  at  $370^\circ\text{C}$  for 24 hours, the resulting waveguide has the refractive index profile  $n(x)$  shown in curve (1) of figure 1. Then we immersed the same glass slide in a dilute melt of 2.5% molar  $\text{AgNO}_3$  in  $\text{NaNO}_3$  at  $380^\circ\text{C}$  for one hour. The resulting waveguide has the refractive index profile shown in curve (2) of figure 1.

On the other hand, a second glass slide is immersed in a dilute melt of 2.5% molar  $\text{AgNO}_3$  at  $380^\circ\text{C}$  for one hour, and the resulting waveguide has the refractive index profile shown in curve(3) of figure 1. These profiles are calculated from the measured values of the propagation constants of the guided TE modes using the well known inverse WKB method<sup>[6]</sup>. A comparison between curves (3) and (2) shows that, for the same depth  $x$  below the surface of the waveguide, the refractive index of the double-ion exchanged waveguide (curve(2)) is less than the refractive index of the single-ion silver exchanged waveguide (curve (3)).

Since the refractive index profile  $n(x)$  is proportional to the  $\text{Ag}^+$  ion

concentration<sup>[4]</sup> in the waveguide, we can conclude that the silver concentration in a previously potassium-exchanged waveguide is less than the silver concentration in a previously unexchanged (blanked) glass slide. This means that the potassium ion halts a subsequent exchange process of the silver ion; as if potassium ions masked the exchange with silver ions.

This masking effect is verified experimentally, at higher temperature; because we repeated the previously described procedure at 425°C. Curves (2) and (3) of figure 2 represent the results at this temperature. Recently, the authors<sup>[9]</sup> published the results of an experimental integrated optical demultiplexer fabricated by the double-ion exchange technique in a glass substrate.

## 2- Theoretical analysis:

It is well known that the ion exchange process is governed by the following partial differential equation<sup>[4], [7]</sup>:

$$\frac{\partial C_i}{\partial t} = \frac{\partial}{\partial x} \left( D \frac{\partial C_i}{\partial x} \right) \quad \text{-----(1)}$$

where  $C_i$  is the concentration of the ion at depth  $x$  (from the surface of the glass slide) and  $t$  is the instant at which we calculate  $C_i$ . The parameter  $D$  is called the "diffusion coefficient" and it is a function of the concentration  $C_i$ . The solution of (1) can be expressed as an infinite power series<sup>[4]</sup> in the form:

$$C_i(x,t) = C_0 [1 - a(x/d) - b(x/d)^2 + c(x/d)^3 + \dots] \quad \text{-----(2)}$$

where  $C_0$  is the ion-concentration at the surface, a, b and c are constants which depend on the relative concentrations and self diffusion coefficients of the species of ions involved in the exchange process<sup>(4)</sup>, since such a process involves at least two types of ions.

The "depth parameter" d is related to the diffusion coefficient of the ion D and the time t by the following relation<sup>(4)</sup>:

$$d = 2\sqrt{Dt} \quad \text{-----(3)}$$

Stewart et.al.<sup>(4)</sup> found that a second-order polynomial is an excellent approximation to the solution of (1) for a single-ion exchange process. Assuming that the refractive index profile is proportional to the concentration  $C_i$  of the ion ( $Ag^+$  in a silver-exchanged waveguide), we can write this profile  $n(x)$  in the form:

$$n(x) = n_s - \Delta n_s [(x/d) + g(x/d)^2] \quad \text{-----(4)}$$

where  $n_s$  is the refractive index at the surface of the waveguide, and  $\Delta n_s$  is the difference between  $n_s$  and the refractive index of the substrate, and g is a constant.

From figures 1 and 2 we see that the refractive index profiles of glass waveguides fabricated by the  $Ag^+$  ion-exchange in a previously potassium-exchanged glass slide - curves (2) in figures 1 and 2 - still have the main characteristics corresponding to a silver-exchanged glass waveguides. So we tried a second-order polynomial approximation for describing the refractive index profile of the double-ion exchanged waveguides in the form  $n(x)$  :

$$n(x) = a_0 + a_1x + a_2x^2 \quad \text{-----(5)}$$



where the a's are unknown coefficients to be determined by fitting to the measured profile using the method of the least squares<sup>[9]</sup>. We obtained an unsatisfactory result, so we had to try a third-order polynomial of the form:

$$n(x) = a_0 + a_1x + a_2x^2 + a_3x^3 \quad \text{-----}(6)$$

where  $a_0 = 1.5856$ ,  $a_1 = -0.00295$ ,  $a_2 = -0.000332$  and  $a_3 = 0.0000146$ .

With these values, the third-order polynomial gives an excellent agreement with the measured profile of curve (2) of figure 2. In our experiments the substrate refractive index  $n_0$  is equal to 1.507 at the He-Ne laser wavelength  $0.6328 \mu\text{m}$ .

Equation (6) allows us to calculate the thickness " $\tau$ " of the waveguide since  $n(\tau) = n_0 = n_s - \Delta n_s$ , where  $\Delta n_s = a_0 - n_0 = 0.0795$  since  $n_s = n(0) = a_0$ .

Substituting in (6) for the a's we obtain:

$$n(\tau) = a_0 + a_1\tau + a_2\tau^2 + a_3\tau^3 \quad \text{-----}(7)$$

which is a third-order equation in  $\tau$ , and can be solved for the waveguide thickness:  $\tau = 20 \mu\text{m}$ . As stated before, since there is a proportionality between the concentration of the  $\text{Ag}^+$  ion and the local increase in the refractive index of the glass slide, we can write the infinite power series solution (2) for the refractive index profile<sup>[4]</sup> as follows:

$$n(x) = n_s - \Delta n_s [C(x/d) + B(x/d)^2 + C(x/d)^3 + \dots] \quad \text{----}(8)$$

where B and C are constants, and "d" is the depth parameter defined by equation (3). Now, from (8) and (6) it is possible to get:

$$a_1 = -\frac{\Delta n_s}{d}, \quad a_2 = -\frac{\Delta n_s}{d^2} B, \quad a_3 = -\frac{\Delta n_s}{d^3} C \quad \text{-----}(9)$$

From (9) we obtain the depth parameter " $d$ " =  $-\Delta n_s/a_1 = 25.4 \mu\text{m}$ .

Using  $d=2(Dt)^{1/2}$ , with  $t=60$  minutes in our experiment, we calculate the effective diffusion constant of  $\text{Ag}^+$  ion in the double-ion exchange process:  $D=2.69 \mu\text{m}^2/\text{sec}$  similarly we determine the other coefficients, B and C, of the infinite power series solution  $B=1.67$  and  $C=-1.47$ .

### 3- Conclusion:

We presented in this paper an experimental and theoretical investigation of planar optical waveguides fabricated by the double-ion exchange technique in glass substrates. We used two consecutive ion-exchange processes: a  $\text{K}^+$  ion-exchange followed by an  $\text{Ag}^+$  one. The masking effect of the  $\text{K}^+$  ion is verified experimentally at two different temperatures:  $380^\circ\text{C}$  and  $425^\circ\text{C}$ . The refractive index profile of the resulting waveguide can be described accurately by a third-order polynomial. A comparison between the coefficients of this polynomial and those of the truncated infinite-power series solution of the diffusion equation allows us to calculate the main parameters of the double-ion exchange process: the depth parameter " $d$ " and hence the effective diffusion coefficient  $D$  of the  $\text{Ag}^+$  ion.



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Figure Captions

Figure 1 : Double ion exchange at 380°C:

Curve (1) : K<sup>+</sup> exchange at 370°C for 24 hours.

Curve (2) : K<sup>+</sup> exchange at 370°C for 24 hours, followed by Ag<sup>+</sup> at 380°C for one hour.

Curve (3) : Ag<sup>+</sup> exchange at 380°C for one hour.

Figure 1 : Double ion exchange at 425°C:

Curve (1) : K<sup>+</sup> exchange at 370°C for 24 hours.

Curve (2) : K<sup>+</sup> exchange at 370°C for 24 hours, followed by Ag<sup>+</sup> at 425°C for one hour.

Curve (3) : Ag<sup>+</sup> exchange at 425°C for one hour.

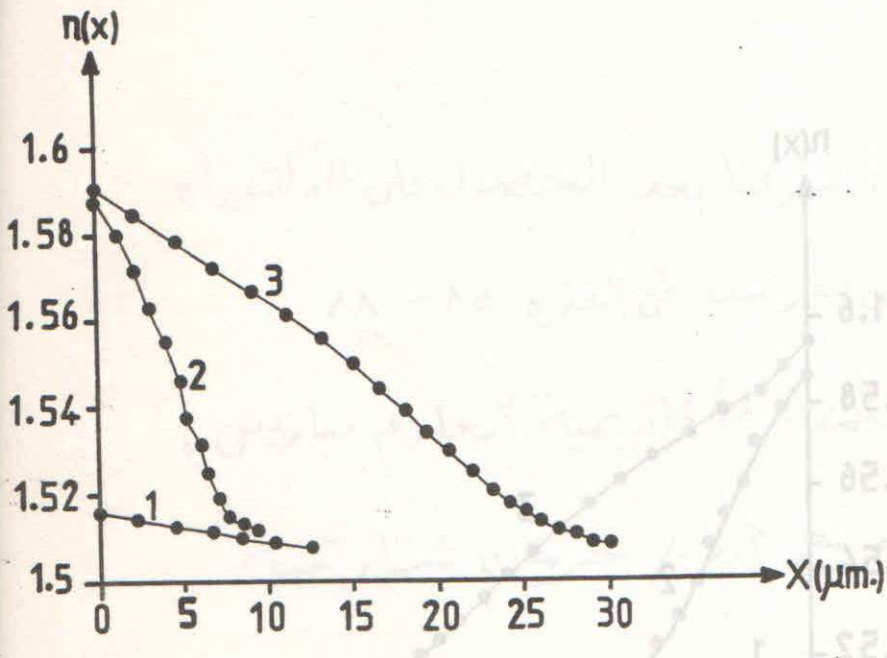


Fig. 1



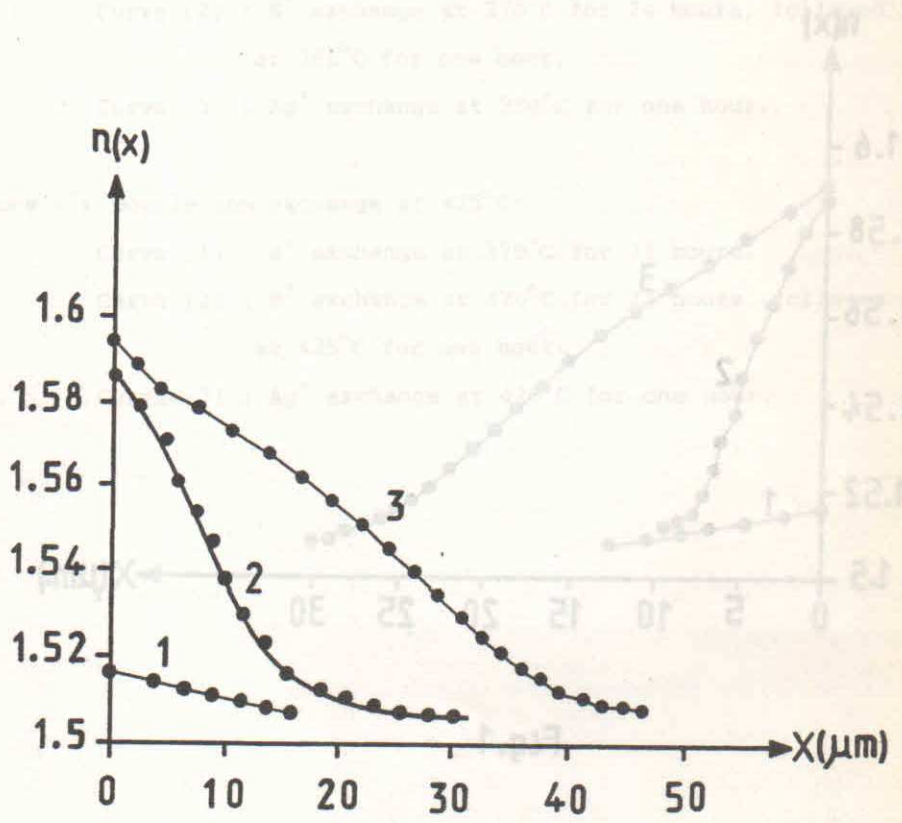


Fig. 2