FORMATION OF OPTICAL WAVEGUIDES IN SPECIALLY DESIGNED Er- AND Yb-CONTAINING SILICATE GLASSES BY Ag^+/Na^+ ION EXCHANGE

LINDA SALAVCOVÁ, FRANTIŠEK ONDRÁČEK*, MARTIN MÍKA**, JARMILA ŠPIRKOVÁ

Department of Inorganic Chemistry, Institute of Chemical Technology Prague, Technická 5, 166 28 Prague, Czech Republic
**Institute of Radio Engineering and Electronics, Czech Academy of Sciences, Chaberská 57, 182 51 Prague, Czech Republic*
***Department of Glass and Ceramics, Institute of Chemical Technology Prague, Technická 5, 166 28 Prague, Czech Republic*

E-mail: salavcol@vscht.cz, jarmila.spirkova@vscht.cz

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Specially designed silicate glasses that contain various rare earth ions have recently become a very promising type of materials for photonics applications (e.g., optical amplifiers or lasers). Here we present a solution to the specific problem in fabrication of planar and channel optical waveguides by $Ag^+ \leftrightarrow Na^+$ ion exchange in Er- and Yb-containing glasses. From our previous experiments it follows that the routinely used conditions of this ion exchange are not suitable for fabrication of optical waveguides that would be used in optical amplifiers. To obtain samples with satisfactory optical properties (particularly, to minimize the optical losses in the waveguides), the total amount of Ag^+ ions in-diffused into the surface layers of the glass has to be lowered. Moreover, the layers should be deepened and the total refractive index increment in the layers should be decreased to achieve the desired change of the refractive index profile. The experiments revealed that lowering of the Ag^+ ions amount in the layers is possible by a substantial decrease of their concentration in the reaction melt. The redistribution of the silver ions in the layers (resulting in further change of the n(x) profile) was achieved by a combination of two fabrication steps - the ion exchange and the annealing (i.e., heating of the as-exchanged layers in air).

INTRODUCTION

In our project we used specially designed silicate glasses [1,2], which have been confirmed to be suitable hosts for erbium and ytterbium ions. These laser-active ions were chosen amongst the rare earths because of their suitable electron configurations (the "three energetic levels system" of pumping and emission [3] and 210 references therein): the Er^{3+} ions are able to emit optical radiation of 1.53 μm (which is close to the mostly exploited wavelength in the telecommunications, 1.55 μm) due to the $^4I_{13/2} \rightarrow {}^4I_{15/2}$ radiative transition; co-doping with Yb^{3+} ions was found to increase substantially the efficiency of Er^{3+} ions´ pumping [3].

Ion exchange process is one of the techniques typically used to form optical waveguides in glass. Most frequently $Ag^+ \leftrightarrow Na^+$ ion exchange is applied as the highly mobile Ag^+ ions (that also cause high refractive index increment) make fabrication of waveguides easier. Our previous experiments revealed that the $Ag^+ \leftrightarrow Na^+$ ion exchange in the currently used high concentrated melts (in our case melt containing 14 mol.% of $AgNO_3$ [4]) results in the formation of optical layers with a relatively high concentration of Ag^+ ions and, consequently, with a high refractive index increment (Δn) in the formed optical layers. However, characteristics of these layers are not satisfactory for their use in the optical amplifiers. First, it is a well-known fact [5] that the silver ions present in the layers are easily photo-reduced to metal form of silver, which then significantly increases optical losses of the waveguides and that is undesirable. From this it follows that in this type of application the amount of in-diffused Ag^+ ions should be minimized (providing that the ratio Ag^+(ion)/Ag^0(metal) is constant). Second, using fabrication process that forms layers with a high Δn indicates that to obtain few-modes planar optical waveguides at 632.8 nm (or, eventually in the application, single-mode channel waveguides at 1.55 μm), the depth of the optical layers has to be very small (even below 3 μm). As the channel waveguides in the optical amplifiers are expected to operate in connection with optical fibres, whose functional radius is approximately 9 μm and typical $\Delta n \approx 0.006$, one can derive that an efficient waveguide-fiber coupling (i.e., low coupling losses of the contact) can not be achieved until the depth of the optical layers is increased and, simultaneously, the refractive index increment is decreased.

In this paper we report on the optimalization of the ion-exchange fabrication process to obtain the waveguides that would be both deeper and less Ag^+ ions containing and that will, eventually, result in formation of low-loss single-mode channel optical waveguides. A set of reaction melts with variable Ag^+ content, in combination with decrease in temperature of fabrication process, was suggested to cut down the concentration of silver ions in the formed layers. Effect of the annealing of the as-exchanged samples on their depth and shape of the refractive index profile was observed as well.

EXPERIMENTAL

Chemical composition of the used silicate glasses that were Na_2O–Al_2O_3–ZnO–SiO_2 matrix with addition of Er_2O_3 and Yb_2O_3 is given in table 1.

Table 1. Chemical composition of the used specially designed glasses.

Glass (wt.%)	Na_2O	Er_2O_3	Yb_2O_3	SiO_2
Er	13.71	2.04	-	67.57
Er-Yb	13.03	1.94	2.00	64.25
Er-2Yb	12.78	1.90	3.92	62.99

The ion exchange (IE) occurred after immersing the pre-cleaned samples into the reaction melt for 4 to 30 min at 230 or 280°C. The used melts were eutectic mixtures of sodium nitrate and potassium nitrate with addition of 1, 2, 7 or 14 mol.% of silver nitrate assigned ANK1, ANK2, ANK7 or ANK14, respectively. The annealing (A), i.e., heating of the as-exchanged samples in the air, was performed at 300°C for 30 min.

Chemical composition of the exchanged layers, i.e., concentration profiles of the exchanged ions, was determined using Scanning Electron Microscopy (SEM). The optical-waveguiding properties of the layers, i.e., number of guided modes and refractive index profiles, were obtained from the optical modes spectra measured by a standard mode spectroscopy (prism coupling set-up) at 632.8 nm by means of inverse WKB approximation.

RESULTS

To examine the effect of the Ag^+ concentration in the melts (and that of change in the reaction temperature) a set of deep multi-mode waveguiding layers was prepared.

The SEM measurement proved (see figure 1a) that using the ANK1 melt in combination with 280°C brought the best results: the amount of the in-diffused Ag^+ ions was lowered substantially (the integral amount decreased to approximately 75 % of the value in the sample fabricated in the ANK14 melt), while the depth of the exchanged layer was almost unaffected. Moreover, this change in the fabrication conditions resulted in lowering Δn (see figure 1b) keeping, at the same time, depth of the optical layer at a relatively high value (compare $n(x)$ profiles of the samples prepared in ANK1 at 280 and 230°C). As the subsequent experiments showed, the ANK1 melt cannot be used for the fabrication of homogeneous waveguides with reproducible properties (due to low concentration of silver ions in the melt, which is not being mixed during the IE); therefore the ANK2 melt was used instead in the following experiments providing homogeneous waveguides, similar to those made in ANK1.

To deepen the optical layers (and, simultaneously, to even more lower the Δn) the annealing process was applied. The strong effect of only 30 minutes of annea-

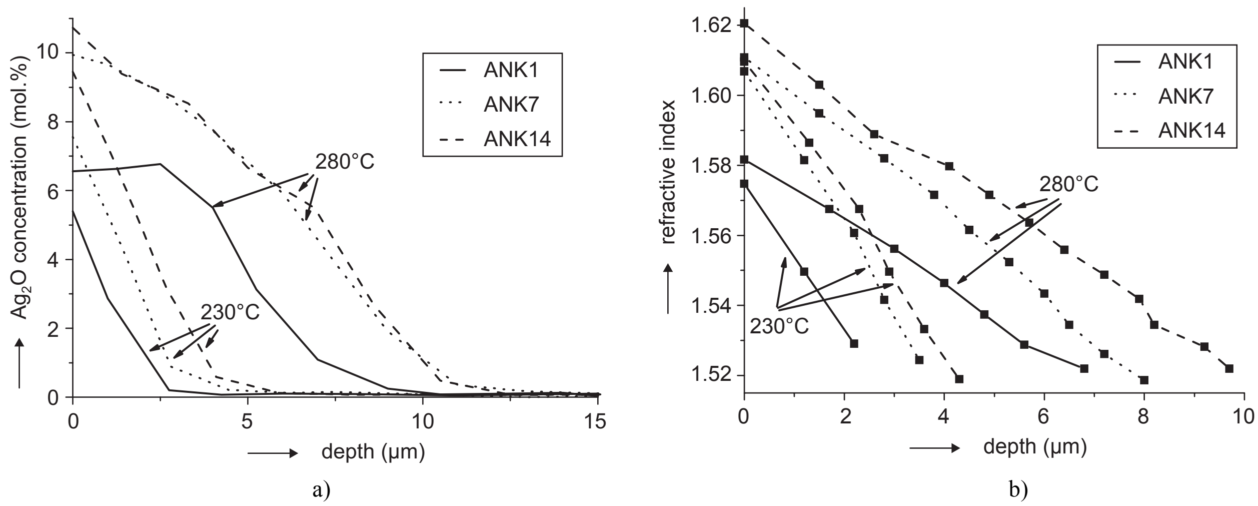


Figure 1. Effect of using various experimental conditions to lower the amount of Ag^+ ions in-diffused into glass on a) concentration profiles of the Ag^+ ions (SEM), b) refractive index profiles (mode spectroscopy) in the formed layers (Er glass, 30 min of IE).

ling on both c_{Ag}(x) and n(x) profiles is presented in figure 2 (these samples, unlike the previous ones, were prepared by a 10-min IE). One can see that the annealing can effectively help to lower the total refractive index increment and deepen the layers at once.

The behaviour of the three used types of glass during the ion exchange (and annealing) differs significantly as the total amount of the rare earth ions, which slow down the rate of migration of the exchanged ions (and also increase the refractive index of the substrate glass), is increasing in the order Er < Er-Yb < Er-2Yb. This effect is shown in figure 3. To obtain similar optical layers in all used glasses, the fabrication conditions have to be modified according to the particular glass.

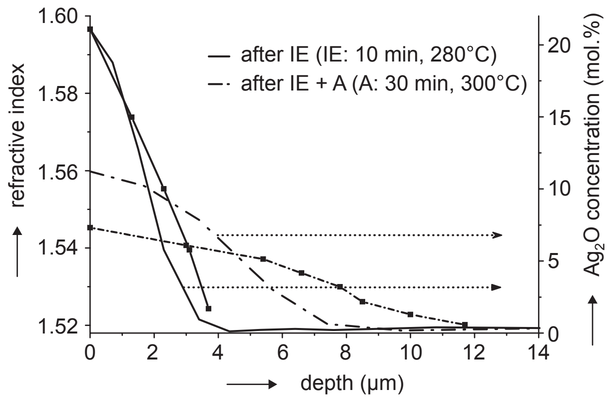


Figure 2. Effect of the annealing on the chemical composition and shape of the refractive index profile in the formed layers (Er glass, ANK2 melt, 10 min of IE).

DISCUSSION

We have found out that to meet the requirements of (i) lowering the amount of Ag^+ ions in-diffused into the glass surface, (ii) deepening the layers and (iii) lowering the Δn in the layers, the combination of the ion exchange from the reaction melt with lower concentration of silver ions and the subsequent annealing is the best choice. A comparison of properties of the samples formed by the former (using ANK14 melt) and the newly suggested fabrication procedure is given in figure 4.

Moreover, using of the annealing process is also desirable in fabrication of the channel waveguides as it helps to modify shape of the channel waveguide (towards the shape that better resembles the optical fiber) and consequently decreases the optical losses of channel waveguide-fiber coupling. The results of measurement of optical losses as well as measurement of the channel waveguides fabricated using the newly designed fabrication procedure will be published elsewhere.

CONCLUSION

In this paper, we report on our research to improve characteristics of optical waveguides that should result in the lowering of optical losses (in the case of planar and channel waveguides) and of coupling losses (in the case of channel waveguides). This improvement can be reached by a modification of the fabrication process (using combination of the reaction melt with low Ag^+ ions content and the annealing process) so that the

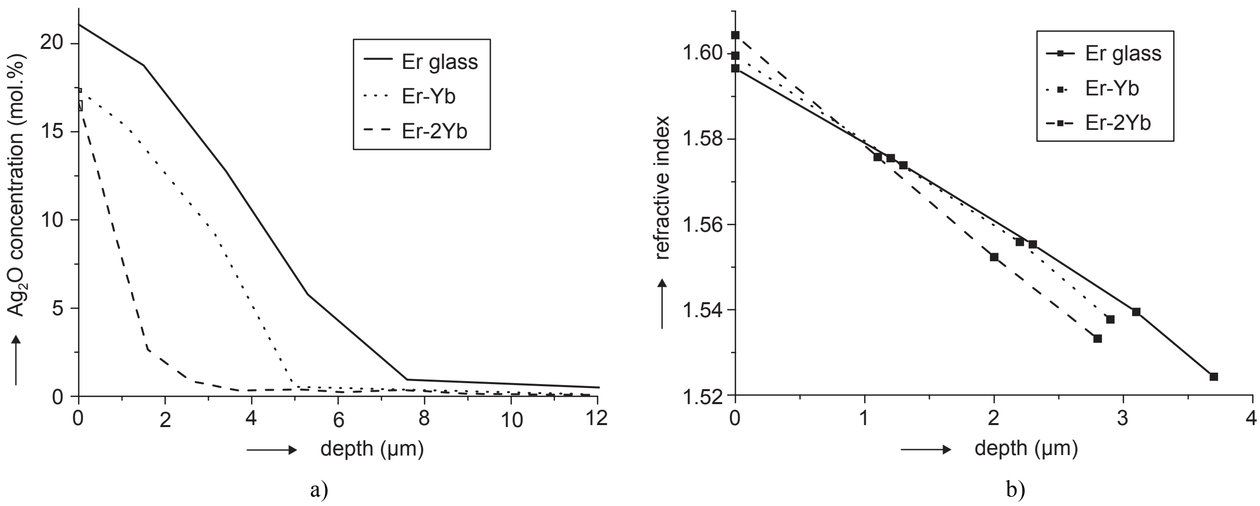
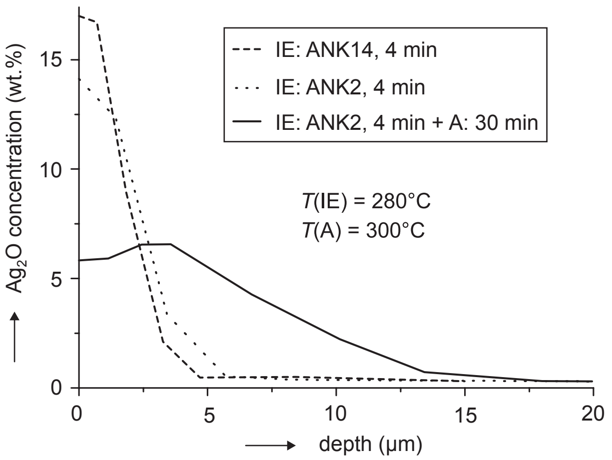
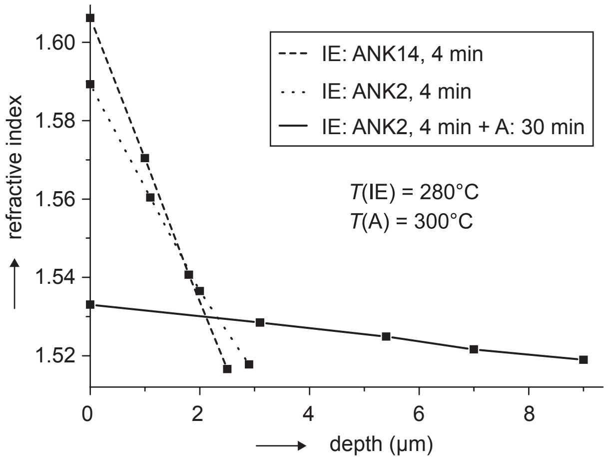


Figure 3. Effect of the total amount of rare earth ions in the glass matrix on the resultant properties of the formed waveguiding layers (ANK2 melt, 280°C, 10 min).

amount of in-difffused silver decreases and the refractive index profile in the layers resembles that of the used optical fibres. The optimal fabrication conditions to obtain few-modes planar waveguides at 632.8 nm (or single-mode channel waveguides at 1.55 µm) are dependent on the type of substrate glass: the ion exchange was carried out in the ANK2 melt at 280°C for 4, 6 and 8 min in Er (2 wt.% of rare earth), Er-Yb (4 wt.%) and Er-2Yb (6 wt.%), respectively; the 30 min. annealing at 300 °C was then applied. By this change in the fabrication procedure we obtained deeper optical layers (from 2.5 µm up to 9 µm, functional radius of the fiber ≈ 9 µm) with a lower refractive index increment (from 0.08967 to 0.01508, Δn of the fiber ≈ 0.006).



a)



b)

Figure 4. Changes in the chemical composition and optical properties of the formed waveguiding layers after modification of the fabrication process (Er glass).

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PŘÍPRAVA OPTICKÝCH VLNOVODŮ IONTOVOU VÝMĚNOU Ag^+/Na^+ V NOVĚ NAVRŽENÝCH SILIKÁTOVÝCH SKLECH OBSAHUJÍCÍCH ERBIUM A YTTERBIUM

LINDA SALAVCOVÁ, FRANTIŠEK ONDRÁČEK*, MARTIN MÍKA**, JARMILA ŠPIRKOVÁ

Ústav anorganické chemie,
Vysoká škola chemicko-technologická v Praze
Technická 5, 166 28 Praha
**Ústav radiotechniky a elektroniky, Akademie věd ČR*
Chaberská 57, 182 51 Praha
***Ústav skla a keramiky,*
Vysoká škola chemicko-technologická v Praze
Technická 5, 166 28 Praha

Nově navržená silikátová skla obsahující ionty různých vzácných zemin se v poslední době stala velmi slibným materiálem pro fotonické aplikace (např. optické zesilovače či lasery). Zde předkládáme řešení specifického problému z oblasti přípravy planárních a kanálkových vlnovodů iontovou výměnou $Ag^+ \leftrightarrow Na^+$ ve sklech obsahujících erbium a ytterbium. Z našich předchozích experimentů vyplývá, že podmínky běžně používané pro tuto iontovou výměnu nejsou vhodné pro přípravu optických vlnovodů, které by měly být použity v optických zesilovačích. Abychom získali vzorky s vyhovujícími optickými vlastnostmi (především pro snížení optických ztrát ve vlnovodech), je nutné snížit celkové množství Ag^+ iontů nadifundovaných do povrchové vrstvy skla. Navíc je třeba prohloubit tyto vrstvy a snížit celkový přírůstek indexu lomu ve vrstvách tak, aby jsme dosáhli požadované úpravy profilu indexu lomu. Experimenty ukázaly, že snížení množství Ag^+ iontů ve vrstvách je možné výrazným snížením jejich koncentrace v reakční tavenině. Redistribuce stříbrných iontů (která má za následek další změnu $n(x)$ profilu) bylo dosaženo zařazením druhého kroku do postupu přípravy - po iontové výměně bylo provedeno žíhání vzorků na vzduchu (tzv. annealing).