Characterization of ion-exchanged waveguides in tungsten tellurite and zinc tellurite Er^{3+}-doped glasses

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Abstract. Among the various materials currently being investigated for further development of Er^{3+}-doped waveguide amplifiers (EDWAs), tellurite glasses are gaining attention because of their broad emission band around 1.5 μm. We report our results on the fabrication of optical waveguides in two kinds of these glasses. Tungsten-tellurite and zinc-tellurite glasses containing sodium and doped with various Er_2O_3 percentages have been prepared and characterized. The Judd-Ofelt analysis was carried out on some of these glasses. Experimentally, broad emission and absorption bands corresponding to transition between $^4I_{13/2} \leftrightarrow {}^4I_{15/2}$ were observed as expected. Lifetimes of the $^4I_{13/2}$ level were also measured. Ion-exchanged planar waveguides were successfully obtained in both types of glasses and characterized by the prism coupling technique; the diffusion process was investigated for the different erbium ion concentrations. All the measured characteristics indicate that both these tellurite glasses are promising for the development of broadband integrated optical amplifiers. [DOI: 10.1117/1.1604782]

Subject terms: tellurite glass; Er^{3+}-doped glass; Er^{3+}-doped waveguide amplifiers; ion exchange; optical waveguides.

Paper AWGE019 received Feb. 27, 2003; accepted for publication Apr. 22, 2003.

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1 Introduction

The current trend toward increasing the capacity transmission in wavelength division multiplexing (WDM) networks makes evident the need for optical amplifiers with a wide and flat gain spectrum in the telecommunication window, and in particular in the C and L bands. Broadening the amplification bandwidth of Er^{3+}-doped waveguide amplifiers (EDWAs) is now a key issue,[1] and tellurite glasses appear to be highly promising materials as they exhibit large stimulated emission cross sections and broad emission bandwidth around the 1.5 μm wavelength. Furthermore, they exhibit a wide transmission range (0.35 to 5 μm), the lowest vibrational energy (about 780 cm^{-1}) among oxide glass formers, low process temperature, good chemical durability, and nonlinear properties.[2-4]

As to integrated optics (IO) fabrication technologies, both for EDWAs and for low-loss passive devices for WDM systems, several different routes have been followed to produce the waveguides onto glass substrates.[5,6] Among them, ion exchange has been recognized as a powerful technique for IO device fabrication[7]: it allows flexibility to accommodate several functions on a glass chip and it is suitable for cost-effective mass production. High optical gain per unit length has already been demonstrated in ion-exchanged EDWAs.[8,9]

Fabrication of waveguides in tellurite glasses has been reported in only a few papers so far,[10,11] though analysis predicts that tellurite EDWAs can exhibit improved gain characteristics.[12] We describe the fabrication and characterization of ion-exchanged planar waveguides in two types of tellurite glasses doped with different Er_2O_3 percentages. The glasses were fabricated, and their stability when changing glass modifiers was assessed and compared. Spectroscopic properties of the glasses were also analyzed and measured. We successfully obtained planar waveguides in both types of glasses. After the ion exchange, the surface quality of the processed samples was investigated to evaluate chemical corrosion. The diffusion process was characterized with a prism-coupling technique for several different erbium ion concentrations. In particular, to the best of our knowledge, this is the first demonstration of ion-exchanged waveguides in zinc-tellurite glasses.

2 Experiment

Two types of tellurite glasses were fabricated and doped with different amounts of Er_2O_3. The first one is a tungsten tellurite, with composition similar to that suggested in Ref. 10, namely containing WO_3, Na_2O, and TeO_2 (WNT glass). The second one is based on ZnO, Na_2O, and TeO_2 (ZNT glass). Table 1 lists the molar composition and the value of the refractive index at 635 nm of the various samples. Chromatic dispersion of the refractive index was also measured by using a semiautomatic instrument developed in-house, named *COMPASSO*, based on a prism-coupling technique. The resulting values of the Abbe number are 20.7 for ZNT and 19.9 for WNT, indicating that both glasses are highly dispersive.

Glass fabrication was similar for both types of tellurite glasses. They were prepared by melting batches composed of analytical grade (Puratronic grade chemicals from Alfa Aesar, MA, USA) of the constituents (TeO_2, WO_3 or ZnO, Na_2CO_3, and Er_2O_3) in a platinum crucible for 2 h at 750°C. WNT glasses were then quenched in air on a stainless steel plate, and the samples were annealed for 2 h at 360°C. Melted ZNT glasses, on the contrary, were cast in a mold, which had been preheated to 270°C, and later annealed for 10 h while gradually cooling down to room temperature. Both glasses were subsequently cut to typical sizes of $1\times1\times0.2$ cm^3 and carefully polished for the optical measurements and waveguide fabrication.

Absorption spectra in the visible and near-infrared regions were measured at room temperature by a double beam spectrometer with a resolution of 1 nm. Emission spectra around 1.5 μm were detected using the 514.5-nm line of an Ar^+-ion laser as an excitation source: luminescence was dispersed using a monochromator with a resolution of 1 nm and detected using a InGaAs photodiode and lock-in technique. Lifetime τ_{exp} of the $^4I_{13/2}$ level was also measured after excitation at 514.5 nm.[13]

Table 1 Molar composition and measured refractive index n ($\pm$0.0005) of the glass samples investigated in this work.

Sample	WO_3	ZnO	Na_2O	TeO_2	Er_2O_3	n@635 nm
WNT0	25		15	60	0	2.0460
WNT005	25		15	60	0.05	2.0471
WNT05	25		15	60	0.5	2.0450
WNT10	25		15	60	1.0	2.0392
WNT15	25		15	60	1.5	2.0356
WNT20	25		15	60	2.0	2.0321
ZNT0		10	10	80	0	2.0452
ZNT1		9	10	80	1.0	2.040
ZNT2		9	9	80	2.0	2.0380

As the transition temperature T_g of these two glasses is relatively low (356 and 290°C for WNT and ZNT glass, respectively), care was taken to perform the ion-exchange processes at temperatures below their T_g. To this purpose, an eutectic solution of $AgNO_3$, KNO_3, and $NaNO_3$ with weight percentages of 2, 43, and 55, respectively, was used, which has a melting temperature of about 245°C. Reagent-grade nitrates were weighted, mixed, and placed in stainless steel crucibles. The ion-exchange temperatures used in this work were 280 and 330°C for WNT samples and 280°C for ZNT ones. Diffusion constants were relatively low, and the $Ag^+ \Leftrightarrow Na^+$ process was preferred to the $K^+ \Leftrightarrow Na^+$, which is much slower. Exchange times necessary to achieve single-mode waveguides at 1.5 μm ranged from 25 min to 6 h, depending on the sample.

Surface quality of the processed samples was visually inspected with a microscope, and the roughness was measured using a surface profiler (Tencor P-10). The effective indices of the waveguide modes were measured at 635, 980, 1310, and 1550 nm using the prism-coupling technique with the *COMPASSO* semiautomatic system, the same instrument already used to measure the refractive indices of the bulk glass samples (reported in Table 1). Finally, the index profile of the waveguides in the ion-exchanged samples was calculated from the measured mode indices using the inverse WKB method.[14]

3 Results and Discussion

3.1 *Glass Characterization: Stability and Spectroscopic Properties*

The glass stability of the two tellurite types, which contain different glass modifiers (Zn or W), was assessed by using the Hruby criterion[15,16]: $K_g=(T_c\text{-}T_g)/(T_m\text{-}T_c)$, where T_g is the glass transition temperature, T_c is the onset crystallization temperature, and T_m is the melting temperature. For WNT glass, $T_g=356$°C, $T_c=390$°C,[10] and T_m is expected to be around 670°C because for glasses, T_m is at about $1.5T_g$[17] (in Kelvin scale); hence $K_g=0.12$. For ZNT glass,[18] $T_g=290$°C, $T_c=420$°C, and $T_m=600$°C; hence $K_g=0.72$. Therefore, according to this criterion, glass stability, which is of primary importance for processing glassy materials, is about six times better in ZNT glass compared to WNT glass.

Absorption cross sections around 1.5 μm were determined from absorption spectra, which consist of sets of $f \rightarrow f$ transitions that can be accounted for by a magnetic or forced electric dipole mechanism. Stimulated emission cross sections were calculated from absorption cross sections and luminescence spectra using the McCumber theory.[19] Figure 1 shows the calculated absorption and emission cross sections for the two types of glasses: these data specifically refer to samples WNT10 [Fig. 1(a)] and ZNT1 [Fig. 1(b)], but the absorption cross section results were practically the same for all the doped samples of the same type. The emission cross sections σ_e of Er^{3+} ions in tellurite glasses are high, since the refractive index n of the glass is high (see Table 1) and the cross section values increase with the refractive index of the host[20] as $(n^2+2)^2/n$. Also the effective emission cross section bandwidth $\Delta\lambda$, defined as $\Delta\lambda=\int\sigma_e(\lambda)d\lambda/\sigma_{e\,\max}$, is very large in tellurite glasses with respect to silicate and phosphate glasses.[3] The values of $\Delta\lambda$ for the WNT and ZNT glasses are 62 and 64 nm, respectively.

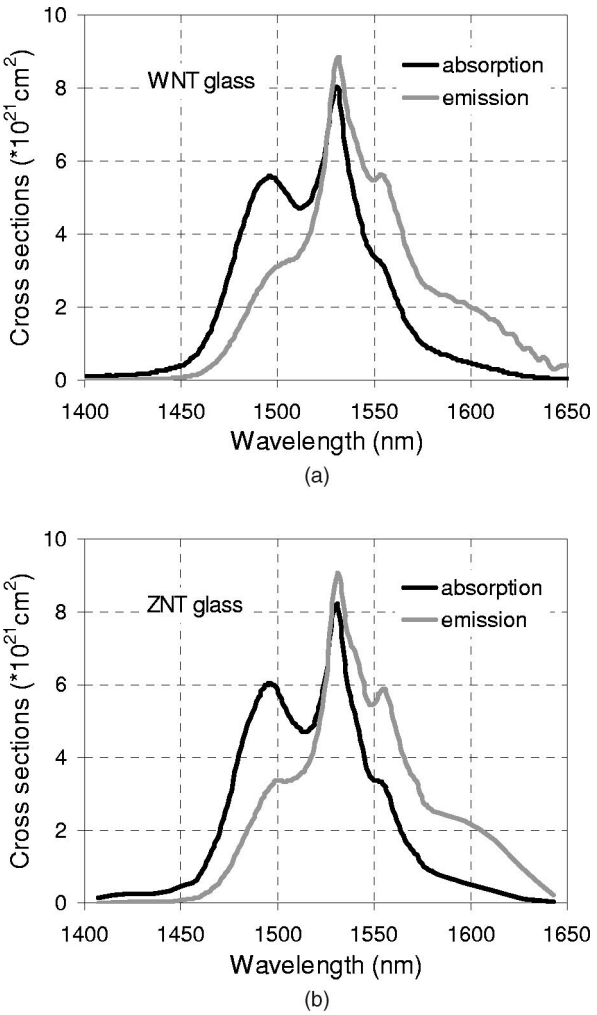

Fig. 1 Absorption and emission cross sections of (a) WNT10 glass and (b) ZNT1 glass.

Table 2 Observed and calculated oscillator strength of Er^{3+} in ZNT1 glass. The ground level of Er^{3+} is $^4I_{15/2}$.

Energy level	Experimental oscillator strengths (10^{-8})	Theoretical oscillator strengths (10^{-8})	
$^4I_{13/2}$	262.26	202.72 (ed)	63.63 (md)
$^4I_{11/2}$	97.41	97.89	
$^4I_{9/2}$	50.36	50.02	
$^4F_{9/2}$	329.09	334.76	
$^4S_{3/2}$	79.77	74.51	
$^2H_{11/2}$	1341.98	1393.75	
$^4F_{7/2}$	251.45	306.41	

Error: $\delta=3.80\times10^{-7}$

Table 3 Calculated spontaneous emission probabilities, branching ratios, and radiative lifetimes for the main emitting states of Er^{3+} in ZNT glass.

Initial level	Final level	Transition wavenumber (cm^{-1})	Transition rate (s^{-1})		Branch ratio (%)	Radiative lifetime (ms)
			E-D	M-D		
$^4F_{5/2}$	$\rightarrow {}^4F_{7/2}$	1661	2.97	2.14	0.08	0.15
	$^2H_{11/2}$	3039	9.12		0.14	
	$^4S_{3/2}$	3793	2.75		0.04	
	$^4F_{9/2}$	6921	273.05		4.11	
	$^4I_{9/2}$	9779	292.54		4.41	
	$^4I_{11/2}$	12030	289.62		4.36	
	$^4I_{13/2}$	15664	2769.66		41.73	
	$^4I_{15/2}$	22159	2994.51		45.12	
$^4F_{7/2}$	$\rightarrow {}^2H_{11/2}$	1378	2.44		0.03	0.12
	$^4S_{3/2}$	2132	0.06		0.00	
	$^4F_{9/2}$	5260	15.7	39.62	0.64	
	$^4I_{9/2}$	8118	314.88		3.64	
	$^4I_{11/2}$	10369	542.74		6.27	
	$^4I_{13/2}$	14003	1156.9		13.37	
	$^4I_{15/2}$	20498	6586.77		76.06	
$^2H_{11/2}$	$\rightarrow {}^4S_{3/2}$	754	0.07		0.00	0.05
	$^4F_{9/2}$	3882	65.24		0.36	
	$^4I_{9/2}$	6740	243.04		1.35	
	$^4I_{11/2}$	8991	174		0.96	
	$^4I_{13/2}$	12625	302.67		1.68	
	$^4I_{15/2}$	19120	17253.88		95.65	
$^4S_{3/2}$	$\rightarrow {}^4F_{9/2}$	3128	1.3		0.03	0.26
	$^4I_{9/2}$	5986	143.16		3.71	
	$^4I_{11/2}$	8237	86.68		2.25	
	$^4I_{13/2}$	11871	1063.11		27.57	
	$^4I_{15/2}$	18366	2562.07		66.44	
$^4F_{9/2}$	$\rightarrow {}^4I_{9/2}$	2858	10.37		0.31	0.29
	$^4I_{11/2}$	5109	59.23		1.75	
	$^4I_{13/2}$	8743	168.16		4.96	
	$^4I_{15/2}$	15238	3152.3		92.97	
$^4I_{9/2}$	$^4I_{11/2}$	2251	2.15	4.74	1.62	2.35
	$^4I_{13/2}$	5885	109.56		25.74	
	$^4I_{15/2}$	12380	309.23		72.64	
$^4I_{11/2}$	$\rightarrow {}^4I_{13/2}$	3634	43.83	22.11	16.37	2.48
	$^4I_{15/2}$	10129	336.86		83.63	
$^4I_{13/2}$	$\rightarrow {}^4I_{15/2}$	**6493.5**	**213.75**	**79.78**	100	**3.41**

The experimental oscillator strengths of the bulk samples were fit on the basis of the Judd-Ofelt parameterization scheme,[21,22] and the phenomenological intensity parameters Ω_i were calculated for both types of glass. Referring in particular to the ZNT glass, the experimental oscillator strengths are compared in Table 2 with the theoretical ones. The calculated values of the intensity parameters Ω_i ($\Omega_2 = 6.11 \times 10^{-20}$ cm^2, $\Omega_4 = 1.75 \times 10^{-20}$ cm^2, and $\Omega_6 = 1.31 \times 10^{-20}$ cm^2) are similar to those already reported for a $19ZnO\text{-}80TeO_2\text{-}1Er_2O_3$ glass[2] and for a $30ZnO\text{-}70TeO_2\text{-}1Er_2O_3$ glass.[23] These values were used to calculate the emission probabilities between the energy levels of erbium in the ZNT host matrix, and then the radiative lifetime τ_{rad} of this emission. The complete data are reported in Table 3.

The experimental lifetimes τ_{exp} were measured by using as an excitation source an argon laser emitting at 514 nm. Figure 2 shows the observed lifetime of the $^4I_{13/2}$ level as a function of Er^{3+} concentration in the two classes of glass. A reduction in lifetime occurs for increasing Er^{3+} concentrations because of concentration quenching, mainly due to a higher probability of nonradiative relaxation due to energy transfer processes. The $^4I_{13/2}$ level decay curves pre-

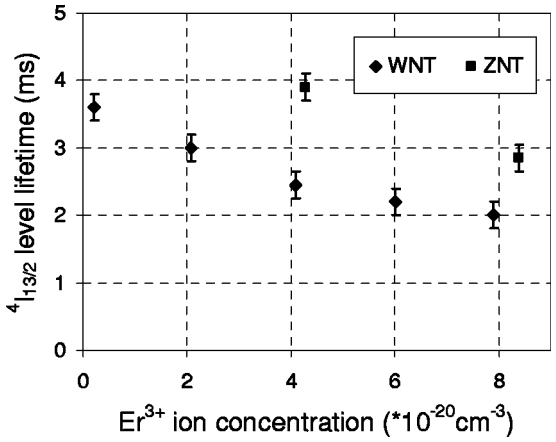

Fig. 2 Measured lifetime of the $^4I_{13/2}$ level as a function of Er^{3+} concentration for the two types of glass.

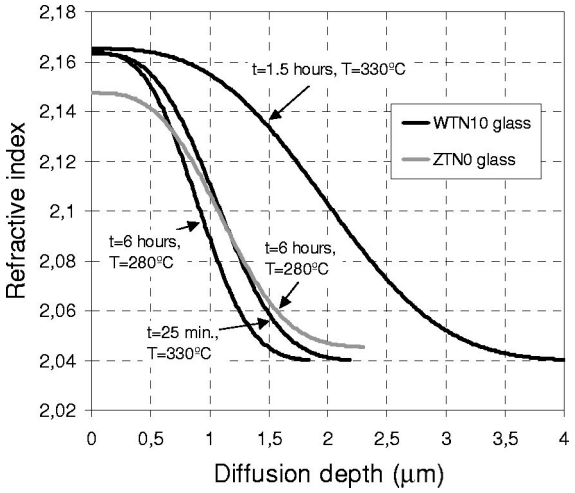

Fig. 3 Calculated index profiles at 635 nm in ZNT0 and WNT10 glass samples for different ion-exchange processes performed in the same molten salt solution.

sented a single exponential decay with values ranging from 3.6 to 2 ms for WNT samples, and from 3.9 to 2.8 ms for ZNT ones. It is evident that, for the same Er^{3+} concentration, lifetime values in ZNT glasses are higher than in WNT glasses. It should also be noted that, for both the ZNT and WNT samples with the lowest concentration of Er^{3+}, we observed an experimental lifetime that was slightly longer than the calculated radiative lifetime. This is possibly due to reabsorption phenomena (samples are about 2 mm thick), and we are now preparing thinner samples (thicknesses of the order of 0.5 mm or less) to verify the dependence of the measured value on the sample thickness. In any case, this issue is worthy of further investigation.

3.2 *Waveguide Characterization*

After ion exchange, visual inspection of processed samples was carried out through an optical microscope: no surface damage was detected in any sample. Surface scanning with a profilometer did confirm that no major changes occurred in the surface roughness due to the process. Subsequently, the presence of guided modes was checked by using a rutile prism to couple light into the waveguides at different wavelengths. The effective indices of TE modes were thus measured.

Multimode waveguides were first fabricated to use the inverse WKB (IWKB) method to determine the refractive index profile resulting from the ion-exchange processes. We obtained up to eight modes (at 635 nm) in WNT glasses and up to three modes in ZNT glasses. Our goal was to characterize the thermal diffusion process of Ag^+ ions in the glasses to be able to design single-mode channel waveguides at 1550 nm. We found out that the analytical function describing the index profile n that better fits the measured effective indices is:

$$n(x) = n_{\text{sub}} + (n_{\max} - n_{\text{sub}})\exp(-x^3/d^3)$$
$$= n_{\text{sub}} + \Delta n \exp(-x^3/d^3),$$

where x is the depth coordinate, n_{sub} is the index of the glass substrate, $n_{\max}$ is the index at the surface, and d is the effective depth of the waveguide. d is related to the diffusion time t (length of the process) by the equation $d = (Dt)^{1/2}$, where D is an effective diffusion coefficient that depends on the molten salt solution, the glass, and the temperature. This type of diffusion profile indicates that the mobility of the incoming alkali ion (Ag^+) is much lower than that of the original ion (Na^+) in the glass. Figure 3 shows the calculated index profiles at 635 nm in WNT10 glass samples for three different ion-exchange processes. From the 1.5-h exchange at 330°C, we concluded that the maximum index change was $\Delta n = 0.126 \pm 0.002$, and that the diffusion coefficient D at 330°C was $9.8 \pm 0.2*10^{-4}$ μm^2/s. Accordingly, single-mode waveguides at 1550 nm should have been obtained—and were indeed obtained—using a 25-min-long ion exchange. If we reduce the exchange temperature to 280°C, 6 h are necessary to produce a rather similar profile, still with one mode at 1550 nm. Diffusion times of 6 h at 280°C were also used to get single-mode waveguides at 1550 nm in ZNT glasses. The index profile of a waveguide in ZNT0 glass is also shown in Fig. 3. In this case, $\Delta n = 0.102 \pm 0.002$ and $D(280°\text{C}) = 7.6 \pm 0.2*10^{-5}$ μm^2/s.

In conclusion, the six samples of WNT glasses, having different Er^{3+} concentrations, were all processed for 1.5 h in the molten eutectic salt solution at 330°C, while the three ZNT samples went through an ion exchange of 6 h at 280°C. The maximum index change and the diffusion depth are plotted in Fig. 4 as a function of Er^{3+} concentration for the two types of glasses. It is worth noticing that in both glasses the index change is not affected by the different Er^{3+} concentrations, which, on the contrary, seem definitely to affect the diffusion depth. With the Er^{3+} concentration increasing up to $8*10^{20}$ ions/cm^3, the diffusion depth for WNT glasses drops from 3 to 1.8 μm (the diffusion coefficient drops from $1.7*10^{-3}$ μm^2/s to $6.0*10^{-4}$ μm^2/s: three times smaller). In ZNT glasses, for a similar change in Er^{3+} concentration, the diffusion depth goes down from 1.28 to 0.72 μm, and again the diffusion coefficient drops by a factor of about three, from

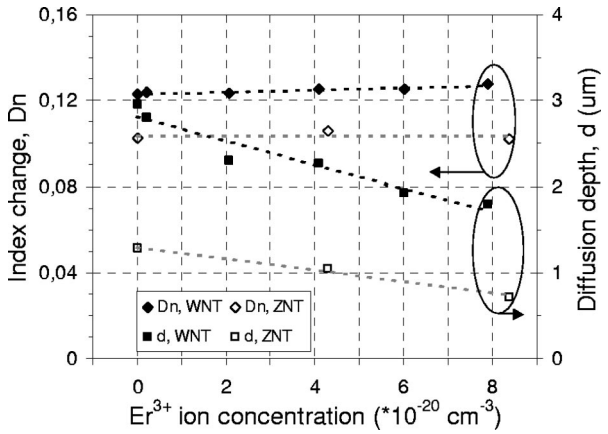

Fig. 4 Index change and diffusion depth as a function of erbium concentration for the two types of glasses. WNT glasses are processed for 1.5 h at 330°C, while ZNT glasses are exchanged for 6 h at 280°C. The molten salt solution used is the same for both glasses.

$7.6*10^{-5}$ $\mu m^2/s$ to $2.4*10^{-5}$ $\mu m^2/s$. Therefore, the decrease of the diffusion coefficients with increasing Er^{3+} concentration is almost the same for the two glasses. A possible reason for this ''slowing down'' of the diffusion process can be related to the increased stability and rigidity of tellurite glass when doped with erbium[18] (or another rare earth, like ytterbium or holmium, for which we observed a similar effect): more stable glass would be more reluctant to lose Na^+ from its structure. Furthermore, the absolute values of D for the two glasses with the same Er^{3+} concentration are almost the same when the ion-exchange process occurs at the same temperature. This fact was observed in the investigated processes at 280°C, even if the Na^+ concentration is 33% lower in ZNT glasses ($\sim 4*10^{21}$ ions/cm^3) than in WNT glasses ($\sim 6*10^{21}$ ions/cm^3). This difference in Na^+ concentration can instead account for the difference in maximum index change—$\Delta n \approx 0.125$ for WNT glasses and $\Delta n \approx 0.103$ for ZNT glasses—which is related to the amount of Na^+ ions that are exchanged by Ag^+ ions at the surface.[10]

Finally, it is important to point out that the waveguide modes obtained in these glasses with the described processes are tightly confined close to the surface (within 2 μm for single mode waveguides at 1.5 μm). Postprocessing, e.g., thermal annealing or waveguide burial, would be necessary for channel waveguides to improve the coupling with standard fibers and to reduce the losses at the air-glass interface.

4 Conclusions

Several samples of zinc-sodium-tellurite (ZNT) glasses and tungsten-sodium-tellurite (WNT) glasses, doped with different amounts of Er_2O_3, are fabricated and characterized. According to the Hruby criterion, zinc tellurite glass shows better glass stability than the tungsten tellurite glass. A broad (wider than 60 nm) emission band around 1.5 μm is observed in all the doped samples, but ZNT samples exhibit longer experimental lifetimes. A deeper investigation of the phenomena affecting the measurement of lifetime, hence of quantum efficiency $\eta = \tau_{exp}/\tau_{rad,}$ in these glasses will be necessary.

Planar waveguides are also successfully fabricated in both types of glasses by an Ag^+-Na^+ ion-exchange technique, using eutectic mixtures to keep the process temperature lower than the transition temperature of the glasses. Characterization of the diffusion process shows that the diffusion depth clearly decreases with increasing Er^{3+} concentration. WNT glasses appear to be more convenient for IO device fabrication because of the shorter (namely, more practical) exchange times necessary to produce a single-mode waveguide at 1.5 μm. This is due to the higher exchange temperature (330 instead of 280°C), made possible by the higher transition temperature of WNT glass ($T_g = 356$°C) with respect to ZNT glass ($T_g = 290$°C).

In conclusion, these two types of glasses exhibit modal and spectroscopic characteristics that, even with their pros and cons, make both of them quite promising for the development of broadband integrated optical amplifiers. Research is in progress to further improve their basic properties and to measure the optical gain in these glasses.

Acknowledgments

This work was partially supported by MIUR, Italy, through the FIRB project, ''Sistemi Miniaturizzati per Elettronica e Fotonica.'' When this work was performed, B. Chen was a visiting scientist at Nello Carrara Institute of Applied Physics, thanks to a TRIL fellowship by ICTP (Trieste, Italy). We are grateful to Mr. Roberto Calzolai for technological assistance.

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Biographies and photographs of the authors not available.