Journal of the Korean Physical Society, Vol. 51, No. 4, October 2007, pp. 1517∼1520

Fabrication and Characterization of Copper-Exchanged BK7 Waveguides

Lei HUANG, Xianfeng CHEN,* Yong YANG and YuxingXIA

Department of Physics, the State Key Laboratory on Fiber Optic Local Area Communication Networks and Advanced Optical Communication Systems, Shanghai Jiao Tong University, 800 Dong Chuan Road, Shanghai 200240, China

(Received 7 September 2006)

In this paper, we report fabrication of a copper-exchanged optical planar waveguide in BK7 glass by using ion exchange with $ZnCl_2$+Cu_2Cl_2 as ion source. The measured loss of the fabricated waveguides was as low as about 0.36 db/cm. The waveguides can hold 2 – 4 modes with 5- to 30-minute exchange and 1 mode with 2- or 3-minute exchanges. The effective refractive index of each mode was determined by using the m-line test system at a wavelength of 0.65 μm. In addition, the cubic spline function interpolation method was employed to recover the gradient refractive index profiles of the copper-containing waveguides. The study on the annealing process for copper-exchanged waveguide is also discussed.

PACS numbers: 42.82.Et, 42.79.Gn
Keywords: Copper-exchanged, Planar waveguides, BK7 glass, Cubic spline function

I. INTRODUCTION

The ion-exchange technique has been considered as an important method for producing high-quality and low-loss graded refractive index optical waveguides, which is very crucial due to their compatibility with optical fibers and the ease of their integration into a system. Ion-exchanging in glass is typically carried out by replacing Na^+ ions with different ions, such as Ag^+, K^+, Cs^+ and Er^{3+} [1], but waveguide formed by ion exchange with copper, especially for Cu^+ have not been studied broadly. An interesting feature of copper ion exchange waveguides, the existence of two relatively stable oxidation states-Cu^+ and Cu^{2+}, cause them to have their own characteristic behaviors. The cooper-containing waveguide has drawn attention recently because of its blue-green luminescence property [2] and its intensity-dependent refractive index, which is caused by an enhancement of the third-order optical nonlinearity of the host glass by the formation of Cu metallic nanoclusters [3].

In this work, copper-containing planar waveguides were fabricated by using an ion exchange process, a substrate of BK7 glass, and a Cu^+ source. Experimental results for the loss, the recovered refractive index profile, and the diffusion process in the Cu^+-containing waveguide were investigated. The annealing process was also studied.

*Corresponding Author: xfchen@sjtu.edu.cn

II. EXPERIMENT

In our ion-exchange experiment, BK7 glass was chosen as substrate and $ZnCl_2$+Cu_2Cl_2 (1:1 mass) as the ion source. The ion source we used contained Cu^+, which has a stronger blue-green emission than Cu^{2+} [4]. $ZnCl_2$ was used to impede Cu^+ from oxidizing into Cu^{2+}.

The ion exchange temperature was 500 °C (see Figure 1) and the processing times was varied from 5 to 30 minutes. The waveguide properties of the samples were tested by means of a m-line spectroscopy technique with a laser at a wavelength of 0.65 μm that was coupled into the waveguide by a ZF7 glass prism. The loss was tested by using digital scattering method [5]. Furthermore, the refractive index profile was recovered by using a cubic spline interpolation method [6], which is a non-destructive method for giving an accurate, smooth refractive index profile.

Moreover, we directly fabricated the single-mode copper-ion-exchanging waveguides by minimizing the exchanging time to 2 or 3 minutes, which is useful in reality. The annealing temperature was 500 °C and the sample was exchanged in a molten liquid for 30 minutes at 500 °C.

III. RESULTS AND DISCUSSIONS

The diffusion of copper from the ion source into the substrate proceeded very quickly so that the changes in

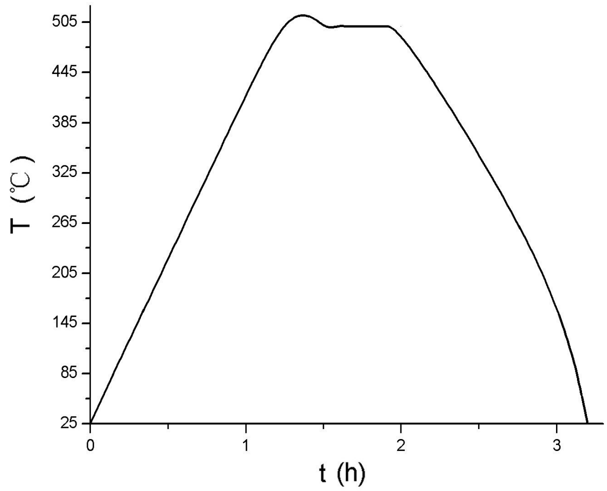

Fig. 1. Temperature in the ion exchanging furnace versus time.

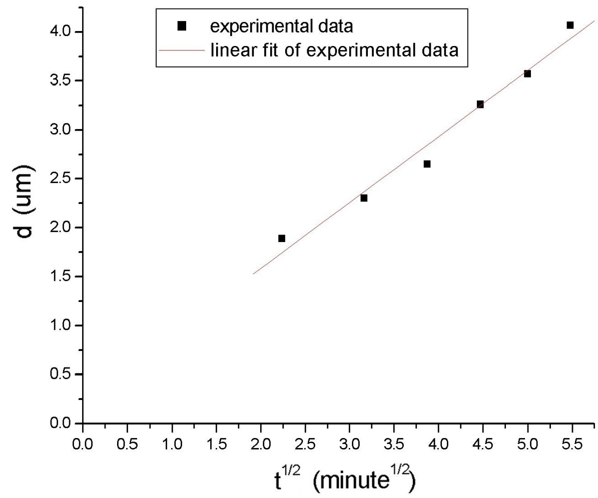

Fig. 2. Diffusion depth versus the square root of the diffusion time.

Table 1. Diffusion depths and changer in the refractive indices of Cu^+-Na^+ waveguides in BK7 glass at a wavelength of 0.65 μm after exchanges of 5, 10, 15, 20, 25, 30 minutes.

t	Number	Δ n(surface)			Diffusion depth(μm)		
(min)	Of mode	TE	TM	Average	TE	TM	Average
5	2	0.0197	0.0171	0.0184	1.930	1.850	1.890
10	2	0.0235	0.0157	0.0196	1.990	2.615	2.302
15	2	0.0159	0.0186	0.0172	2.560	2.740	2.650
20	2	0.0196	0.0169	0.0182	3.445	3.080	3.262
25	3	0.0207	0.0243	0.0225	3.550	3.590	3.570
30	3	0.0185	0.0171	0.0178	4.065	4.069	4.067

the substrate could be easily identified after several minutes. In our experiments, 5, 10, 15, 20, 25 and 30 minutes were chosen as exchange times to fabricate 6 samples. We define the diffusion depth to be the depth of the half height of the refractive index profile. The results are shown in Table 1. In Table 1, we find that after the ion exchange process, the surface refractive index increases. The increase in the surface index is about 0.018. The exchange process is caused by the difference in the concentration of Cu^+ between the glass and the ion source.

There is a relationship between the diffusion depth and the diffusion time,

$$d = \sqrt{D(T)t}, \tag{1}$$

where d is the diffusion depth, t is the exchange time and D(T) is the diffusion constant at a certain temperature T. As Figure 2, shown a linear fit is done on our experimental data and we find that it satisfies the equation very well except that it doesn't pass through the origin. The possible reason is that there still remain some solutions on the substrate when the waveguide has been taken out of the ion source. The diffusion constant calculated from the linear fit is

$$D(T = 500\ ^\circ\mathrm{C}) = 0.756 \times 10^{-14}(\mathrm{m}^2/\mathrm{s}). \tag{2}$$

The diffusion constant is a relatively large one compared to the value of 5.39×10^{-16} (m^2/s) in Ref. 7, in which the experiment was done at a temperature of 350 °C and an applied field of a 30 V/mm, However, the diffusion constant is smaller than that of the Cu^{2+} exchange using $CuSO_4$+Na_2SO_4 as ion source at a temperature of 580 °C, whose diffusion constant is $D(T = 580°\mathrm{C}) = 1.214 \times 10^{-14}(\mathrm{m}^2/\mathrm{s})$ [8].

Cubic spline function interpolation, proposed by us [6], is a method that can recover the refractive index profile smoothly. It is employed to recover the refractive index of the copper-containing waveguide fabricated by us, as shown in Figure 3. Clearly, the longer the exchange time, the deeper the Cu^+ exchange into the substrate.

The part essential of the annealing process is the second diffusion. In this case, the ion source is ions that have diffused into the substrate in the first ion-exchanging process. In the experiments, the waveguide exchanged for 30 minutes was used as the sample to be annealed for 45 and 90 minutes; then, the refractive index profile was recovered to compare the difference between the sample before and after annealing. From Figure 4, it is clear that the surface refractive index decreases with increasing annealing time and that the refractive index profile becomes flatter. Meanwhile, the diffusion depth also increases. During the annealing process, the total number of ions is unchanged, and the ions in the glass will keep on diffusing because of the concentration gradient at high temperatures, so the profile becomes flatter during annealing process in our experiments, which agrees well with the theoretical prediction.

In our experiments, we find that the effective indexes for the TE and the TM modes are different, which shows that the waveguides become anisotropic. This result dif-

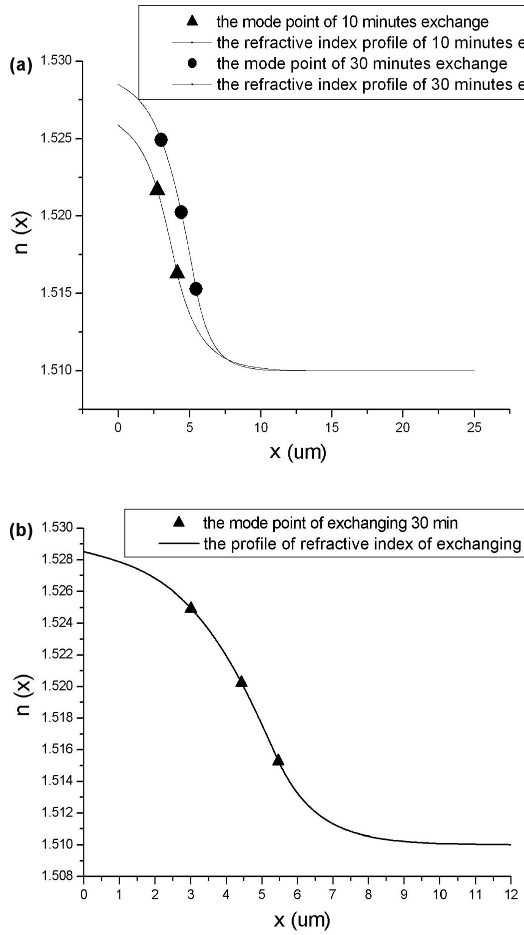

Fig. 3. Refractive index profile of the waveguide exchange for 10 minutes and 30 minutes, as recovered by using cubic spline function interpolation method of the TE mode at 0.65 μm.

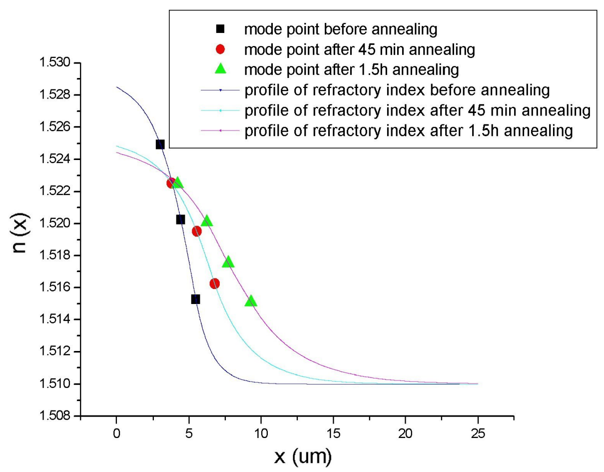

Fig. 4. Refractive index profile of TE mode at a wavelength of 0.65 μm of a waveguide exchanged for 30 minutes and of that after annealing for 45 or 90 minutes.

fers from that reported by P. Nebolova *et al.* [9]. As we

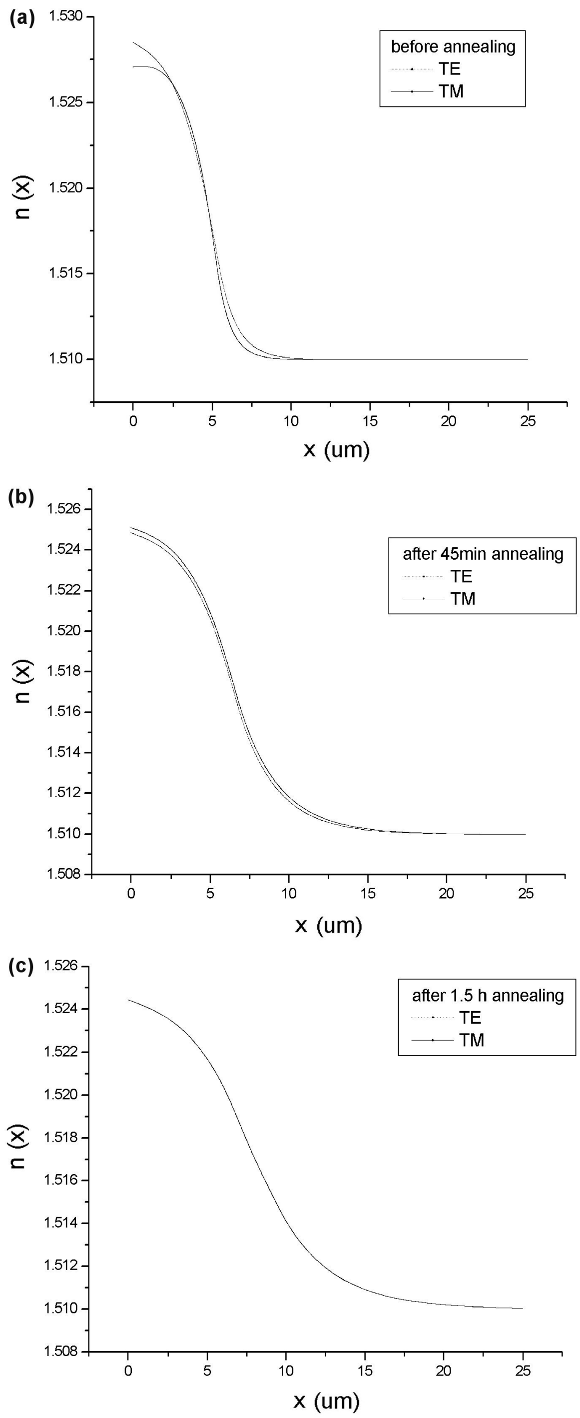

Fig. 5. Refractive index profiles of the TE and the TM modes at a wavelength of 0.65 μm (a) a waveguide exchanged for 30 minutes of those after an annealing for (b) 45 minutes or (c) 90 minutes.

know, the ionic radius of Cu^+ (96 pm) is almost same as that of Na^+ (95 pm). In BK7 glass, there is also K^+ whose ionic radius is about 133 pm, which will also exchange with Cu^+. The difference in ionic radii will result in a birefringence anisotropy [10,11]. After annealing, the birefringence anisotropy will disappear and the waveguide will be isotropic again. This process is shown in Figure 5. The equilibrium position of the ion brings an equilibrium of the polarization. As a result, annealing for a long time leads to an isotropic copper-exchanging waveguide.

The loss of the waveguide exchanged for 30 minutes was about 0.36 db/cm. After the annealing process, the loss will decrease. After annealing for 90 minutes, the loss was measured to be as low as 0.31 db/cm.

Also, after annealing for 3 or 4 hours, the 2-mode waveguide will convert to a single-mode one whose loss is lower than that of the direct fabricated single-node waveguide.

IV. SUMMARY

Copper-exchanged planar waveguides were fabricated on substrates of BK7 glass with $ZnCl_2+Cu_2Cl_2$ as an ion source by using an ion exchange process. Samples were optically characterized by using m-lines spectroscopy. The refractive index profile was determined by using the cubic spline function interpolation recovery method. For BK7 glass as a substrate and $ZnCl_2+Cu_2Cl_2$ as an ion source at 500 °C, the diffusion constant of Cu^+ was $0.756 \times 10^{-14}(m^2/s)$. The exchange of Cu^+ in the BK7 glass led to a birefringence anisotropy, though it disappeared after the sample had experienced an annealing process for a long enough time. By varying the exchanging time and the annealing time, we obtained a prospected refractive index profile. The loss of the copper-exchanging waveguide was about 0.36 db/cm and became even smaller after an annealing process. Generally, the experimental results indicate that copper-exchanging in BK7 glass in $ZnCl_2+Cu_2Cl_2$ molten liquid is an easily controllable way to fabricate applicable Cu^+-containing waveguides.

ACKNOWLEDGMENTS

This research was supported by the National Natural Science Foundation of China (No. 60477016 and No. 10574092), the Foundation for Development of Science and Technology of Shanghai (No. 04DZ14001) and the "Shu-Guang" Scholar Plan of Shanghai Education Committee.

REFERENCES

[1] R. V. Ramaswamy and R. Srivastava, J. Lightwave Technol. **6**, 984 (1988).
[2] R. Debnath. J. Lumin. **43**, 375 (1989).
[3] P. Mazzoldi, G. W. Arnold, G. Battaglin, F. Gonella and R. F. Haglund, J. Nonlinear Opt. Phys. Mater. **5**, 285 (1996).
[4] J. Spirkova, P. Tresnakova-Nebolova and M. Mika, Optical Materials **25**, 101 (2004).
[5] Y. Jiang, Z. Cao and Q. S. Shen *et al.*, Acta Optica Sinica **19**, 1142 (1999).
[6] W. Liao, X. Chen, Y. Chen and Y. Xia, J. Optical Soc. American A **22**, 1334 (2005).
[7] H. Marquez, D. Salazar, A. Villabos *et al.*, J. Appl. Opt. **34**, 5817 (1995).
[8] W. Pengfei, Z. Jie, E. Shulin and G. C. Righini, Chinese J. Lasers **31**, 10 (2004).
[9] P. Nebolova, J. Spirkova, V. Perina, I. Jirka, K. Mach and G. Kuncova, Solid State Ionics **141-142**, 609 (2001).
[10] F. Gonella, F. Caccavale, L. D. Bogomolova, F. D'Acapito and A. Quaranta, J. Appl. Phys. **83**, 1 (1998).
[11] D. Salazar, A. Villalobos, H. Porte, L. J. Villegas and F. Marques, J. Mod. Opt. **46**, 657 (1996).