APL accepted 1937.

# Buried laser waveguides in neodymium-doped BK-7 by $K^+$-$Na^+$ ion-exchange across a direct-bonded interface.

Corin B. E. Gawith, Tajamal Bhutta, David P. Shepherd, Ping Hua, Ji Wang, Graeme W. Ross, and Peter G. R. Smith.

Optoelectronics Research Centre
University of Southampton
Southampton
S017 1BJ
United Kingdom

Telephone: +44 1703 593172
Facsimile: +44 1703 593149
Email: cbeg@orc.soton.ac.uk

## Abstract

We report a technique for producing single-step buried $K^+$-$Na^+$ ion-exchanged waveguide lasers in neodymium doped BK-7. Direct bonding is the basis for this process, providing atomic contact between two chemically modified BK-7-type substrates followed by a 350 °C treatment suitable for simultaneous annealing and intersubstrate ion-exchange. Characterization of a 6-mm long device was performed using a Ti:Sapphire laser operating at 808 nm. The resultant laser output exhibited TE polarized single-spatial-mode operation with losses of < 0.4 dB $cm^{-1}$ and a maximum output power of 8.5 mW for 249 mW of absorbed pump power.

Ion-exchange techniques provide a simple and versatile method of creating optical-fibre-compatible planar waveguide devices in glass. Actively studied and reviewed[1,2] for many years, the ion-exchange process remains almost exclusively based upon the use of a molten salt bath as a source of alkali ions, with applied heat or electric fields providing control over the rate of exchange of ions with the substrate material. The few exceptions to this method include depositing a thin layer of metal onto a substrate, a technique mainly used in the fabrication of silver and copper ion-exchanged waveguides[2], to act as an anode for field-assisted ion-exchange processes.

The production of efficient integrated optics devices in glass requires that the inherent losses caused by light scattering from surface imperfections at the waveguide interface must be overcome, a problem leading researchers to investigate novel ways of burying waveguides below the surface of an ion-exchanged substrate. This effect is generally achieved by diffusing the waveguide region further into the material[3] with a secondary heat or electric field treatment, or alternatively by means of a second ion-exchange process designed to lower the refractive index of the substrate at the surface[2]. The additional improvement of waveguide symmetry can also reduce fibre-coupling losses[1], but care must be taken to account for the further diffusion of ions in the material, which can alter the waveguide depth and refractive index profile.

A more recently applied method, to avoid surface related losses in borosilicate glass, has been to bond a second layer onto an ion-exchanged substrate[4], burying the waveguide completely. This is performed using a combination of Van Der Waals forces (inter-atomic forces present when two flat bodies are contacted) and suitable chemical and heat treatments, which together comprise the Direct Bonding[5] (DB) technique. Such a process can be used to create low-loss, seamless, vacuum tight bonds between dissimilar material layers with no deleterious modification[6] of the original substrates, as evidenced in the design and realisation of various multi-layered active waveguide devices[7,8].

In this letter we describe our initial study towards producing a buried waveguide laser in neodymium doped BK-7 glass by ion-exchange across a direct bonded interface. In contrast to the methods previously described, we have used the DB technique to provide a region of atomic contact between chemically modified BK-7 substrates, altered to create opposing potassium and sodium concentration gradients across the bonded interface, between which ion-exchange can take place. By taking this approach we have acheived a single-step buried ion-exchange mechanism with intrinsically low waveguide losses. It is the annealing phase used in DB that provides the key to this process, as the time and temperature used to strengthen direct bonded BK-7 substrates (350-400°C for several hours) is also appropriate for $K^+$-$Na^+$ ion-exchange. By performing these processes simultaneously we remove any risk of secondary diffusion of the waveguide layer, or damage to the optically polished substrates (surface condition is a critical prerequisite for DB), whilst also escaping the hostile environment associated with a molten salt bath.

Preparation of the rare-earth-doped substrate (illustrated as the top layer in Fig. 1) used in this experiment began by mixing pieces of commercially available BK-7 glass with 2 wt.% of neodymium oxide in a ceramic crucible. This mixture was placed in an electrically heated furnace and stirred to promote uniformity of neodymium ions within the host material. A temperature range of 850 °C to 1450 °C was available throughout the doping process, the melt being fined at 1400 °C and removed from the furnace at 1300 °C. Cast into a stainless steel mould, the melt was finally annealed in a muffle furnace at 580 °C. Further details of this technique can be found in Ref. 9. The second glass type, which forms the cladding layer of the device (the lower level in Fig. 1) and acts as a source of potassium for ion-exchange, was prepared from basic oxide materials by a similar technique. Based on the chemical composition of commercially available BK-7 [3], our oxide mix was altered to allow an additional 4 wt.% (approx.) of $K_2O$ into the glass by removing an equal amount of $Na_2O$. Such an offset in glass composition between our BK-7-type and neodymium doped substrates, the latter of which features 6 wt.% of both $K_2O$ and $Na_2O$ (this is slightly lower than normal due to the doping process), allows the realisation of buried laser waveguides by internal ion-exchange processes in DB structures. Multiple samples have been produced by this method, proving that this is a robust technique, but it should be noted that the chemical composition of each substrate layer has yet to be optimised.

From each glass type a 2 mm thick substrate, of 10 mm × 5 mm surface area, was diced and polished to provide an optically flat surface suitable for DB. After cleaning, a mixture of $H_2O_2$-$NH_4OH$-$H_2O$ (1:1:6), followed by several minutes of rinsing in deionised water, was applied to both materials in order to render their surfaces hydrophilic[10]. The doped and undoped layers were then brought into contact at room temperature and finger pressure applied in order to promote adhesive avalanche[11]. This effect forces any excess air or liquid from between the two substrates, as demonstrated by the removal of all interference fringes across the 10 mm $\times$ 5 mm bonded interface. Annealing of the sample at 350 °C for 6 hours provided ample bond strength for further machining and sufficient ion-exchange to create a single-mode waveguide on the neodymium-doped side of the bonded interface. The sample was then diced and polished to the approximate absorption length expected for our neodymium doped waveguide (~ 6 mm). Dimensions of the resulting buried laser waveguide device are given in Fig. 1.

The discovery of a buried waveguide near the bonded interface of our sample prompted several tests to confirm its origin as an internal ion-exchange mechanism. Optical testing produced no evidence of waveguiding as a result of any pre-annealing processing step (from polishing and cleaning to the DB of the two layers), although every annealed sample exhibited a buried waveguide. This result indicates that the DB heat treatment is directly responsible for the creation of optical confinement within our device.

Confirmation of the ion-exchange process within our direct bonded samples was provided by a scanning electron microscopy (SEM) based compositional line profile analysis (LPA). This technique was used to measure the difference in relative concentration for potassium and sodium across the polished end-faces of an annealed device and an unannealed control sample, the results of which are given in Fig. 2. As illustrated by the graph, there is a significant ionic redistribution between the line profiles of the unannealed (broken line) and annealed (solid line) samples, wherein $K^+$ and $Na^+$ can be seen to interdiffuse across the bonded interface. This heat-related change in chemical composition provides proof of an internal ion-exchange mechanism within our devices, the result of which is a single-step buried waveguide. Unfortunately, the low resolution available from the SEM, most evident in the rounding of the predicted step function of material composition between the two unannealed layers, requires that a mathematical deconvolution be performed in order to isolate the overall changes in ionic distribution from our results. As such our future plans include a more accurate secondary ion mass spectroscopy (SIMS) compositional analysis, combined with further studies into the effects of annealing time and temperature on waveguide parameters, in order to enhance our diffusion modelling.

Characterisation of spectroscopy, laser performance, and propagation loss of the device was performed with a tunable (700 nm to 850 nm) Ti-Sapphire laser. Pump radiation was focussed for launch into the waveguide by means of a ×10 microscope objective to a measured input spot size of approximately 3 μm. Optimisation of the launch was performed in relation to the fluorescence measured by a silicon detector, and a combined launch and absorption efficiency of 52 % was calculated from measurements of input and output pump power from the waveguide. Flourescence spectra from the waveguide and bulk neodymium-doped BK-7 were typical of similarly produced glass[9], as was the recorded flourescence lifetime of 470 μs. The typical setup used for waveguide characterisation is given in Fig. 3.

Laser action was achieved in the $Nd^{3+}$ $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition by butting plane mirors to the end faces of the device and pumping the waveguide at 808 nm. Thresholds as low as 21 mW of absorbed power were obtained with these devices using two HR mirrors. With a 3 % output coupler the lasing threshold rose to 41 mW absorbed power and a slope efficiency of 6 % was obtained. Maximum output power with this configuration was 8.5 mW ($\lambda$ = 1059 nm) for an absorbed power of 249 mW, although no effort was made to optimise the overlap of the pump and signal radiations. The output of the laser waveguide remained TE polarized regardless of pump polarisation, a result confirmed in several similar devices. Such an effect could be caused by the unusual combination of stresses in our device, resulting from both the ion-exchange and DB processes, although this has yet to be investigated.

Mode profiling of the output from the buried waveguide was performed with a silicon camera and PC based evaluation software. It was observed that both the laser output and pump throughput were in the fundamental spatial-mode, with guided output spot sizes ($1/e^2$ intensity radius) of 3.3 μm and 2.5 μm respectively. The bonding process was not designed to give confinement in the horizontal plane, and the laser mode was observed to have an unguided spot size of ~ 40 μm.

Propagation loss in the waveguide was estimated by investigating the relationship between laser threshold and output coupling[12]. Assuming negligible lower laser level population, constant loss with variable pump intensity, and constant laser mode size, the threshold of the laser ($P_{th}$) is expected to obey the equation:

$$P_{th} = k\,[2\alpha l - \ln(R_1 R_2)]$$

where k is a constant encompassing the pump and laser mode spatial properties and material parameters, l is the length of the monolithic cavity, $R_1$ and $R_2$ are the mirror reflectivities and $\alpha$ is the propagation loss coefficient. By varying the output coupler a plot of threshold against $-\ln(R_1R_2)$ was produced (Fig. 4), the x-axis intercept of which gives an estimate of propagation loss. For our buried waveguide a value of 2.4 $m^{-1}$ was measured for $\alpha$, corresponding to a loss of 0.1 dB $cm^{-1}$, although the availability of suitable mirror sets limited these results to just three points on the graph. However, separate calculations of the expected threshold and slope efficiencies (using the measured mode sizes and previously reported emission cross-sections[9]) lead to values of propagation loss in the range 0.1 dB $cm^{-1}$ to 0.4 dB $cm^{-1}$, a result which compares to the losses achieved by alternative potassium ion-exchange processes in neodymium-doped BK-7 glass[9].

In conclusion, we have reported a fabrication technique for the single-step production of buried $K^+$-$Na^+$ ion-exchanged laser waveguides in neodymium-doped BK-7 glass. The key to this process is the direct bonding technique which provides atomic contact between the two substrate layers and a 350 °C heat treatment suitable for simultaneous annealing and inter-substrate ion-exchange. Opposing chemical gradients of potassium and sodium across the bonded interface were provided by modifying the composition of the cladding material from that of commercially available BK-7 glass. It has been shown that replacing 4 wt.% of $Na_2O$ with $K_2O$ in the cladding glass is sufficient for the production of a single-mode waveguide in an adjoining neodymium doped BK-7 substrate. Characterisation of laser performance from the buried waveguide gives a propagation loss of < 0.4 dB $cm^{-1}$ for the TE polarised laser output. These initial results suggest that optimisation of glass composition and heat treatments could lead to efficient low-loss buried waveguide devices for use with integrated optics. The potential for bonding large wafers of material (to be diced into many smaller devices) for the mass production of low-loss ion-exchanged waveguides could also prove ideal for fabricating laser waveguides with long absorption lengths, such as erbium-doped devices. More generally, the direct bonding technique offers promise in providing extra degrees of design freedom for ion-exchanged waveguide devices.

The Optoelectronics Research Centre is an interdisciplinary research center supported by the Engineering and Physical Sciences Research Council (EPSRC). Corin Gawith and Tajamal Bhutta acknowledge studentships from the EPSRC. The authors thank James Wilkinson for his advice during sample analysis.

**Figure Captions:**

Fig. 1. Schematic diagram of the buried laser waveguide.

Fig. 2. A graph of chemical composition versus distance across the interface for an annealed device (solid line) and an unannealed control sample (broken line). The change in chemical profile with annealing indicates $K^{+}$-$Na^{+}$ ion-exchange between the two glass substrates.

Fig. 3. Experimental arrangement for laser characterisation of the buried waveguide. A pump wavelength of 808 nm was used to obtain a 1059 nm laser output from our device.

Fig. 4. Dependence of laser threshold power with output coupling.

## REFERENCES


[1] R. V. Ramaswamy and R. Srivastava, J. Lightwave Tech. **6**, 984 (1988).

[2] N. V. Nikonorov and G. T. Petrovskii, Glass Phys. and Chem. **25**, 16 (1999).

[3] C. Ciminelli, A. D'Orazio, M. De Sario, C. Geradi, V. Petruzelli, and F. Prudenzano, Appl. Opt. **37**, 2346 (1998).

[4] S. Pelissier, F. Pigeon, B. Biasse, M. Zussy, G. Pandraud, and A. Mure-Ravaud, Opt. Eng. **37**, 1111 (1998).

[5] J. Haisma, B. A. C. M. Spierings, U. K. P. Biermann, and A. A. van Gorkum, Appl. Opt. **33**, 1154 (1994).

[6] K. Eda, M. Sugimoto, and Y. Tomita, Appl. Phys. Lett. **66**, 827 (1994).

[7] C. Gawith, D. P. Shepherd, J. A. Abernethy, D. C. Hanna, G. W. Ross, and P. G. R. Smith, Opt. Lett. **24**, 481 (1999).

[8] C. T. A. Brown, C. L. Bonner, T. J. Warburton, D. P. Shepherd, A. C. Tropper, and D. C. Hanna, Appl. Phys. Lett. **71**, 1139 (1997).

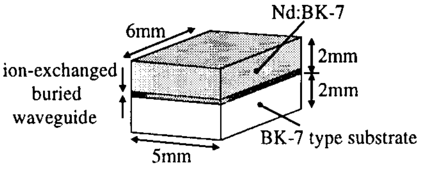
[9] E. Mwarania, J. Wang, J. Lane, and J. S. Wilkinson, J. Lightwave Tech. **11**, 1150 (1993).

[10] G. A. C. M. Spierings, J. Haisma, and T. M. Michielsen., Philips J. Res. **49**, 47 (1995).

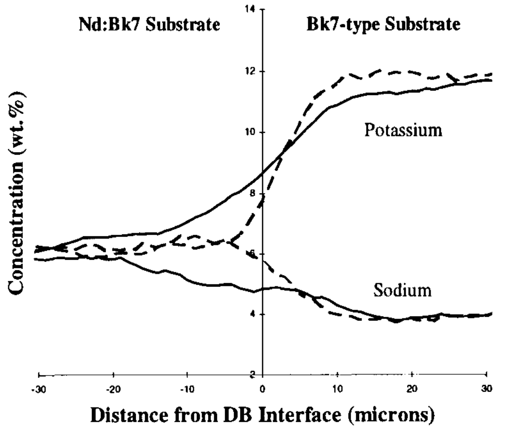
[11] J. Haisma, G. A. C. M. Spierings, T. M. Michielsen, and C. L. Adema, Philips J. Res. **49**, 23 (1995).

[12] D. Findlay and R. A. Clay, Phys. Lett. **20**, 277 (1966).

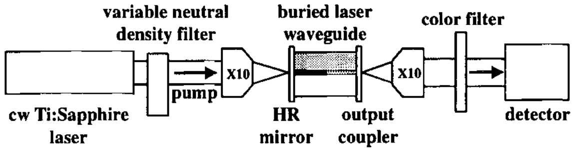
Corin Gawith, Paper Ref: #L99-2313, Figure 1 (TOP).



Corin Gawith, Paper Ref: #L99-2313, Figure 2 (TOP).



Corin Gawith, Paper Ref: #L99-2313, Figure 3 (TOP).



Corin Gawith, Paper Ref: #L99-2313, Figure 4 (TOP).