

# Amplification from Nd<sup>3+</sup> doped Si–Ti sol–gel channel waveguides

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## ARTICLE INFO

### Article history:

Available online 6 December 2008

### PACS:

42.60.Da

42.70.–a

42.82.Gw

### Keywords:

Neodymium

Sol–gel waveguide

Amplifier

Grating coupler

Up-conversion

## ABSTRACT

We report the sol–gel chemistry reaction system and processes used to realize the first grating coupled monolithic solid state optical amplifier based on a Nd<sup>3+</sup> doped sol–gel tapered rib waveguide. The sol–gel matrix composition is 50SiO<sub>2</sub>:50TiO<sub>2</sub>:10AlO<sub>1.5</sub>:1NdO<sub>1.5</sub>. The signal (1064 nm) and pump (803 nm) beams were coupled via grating couplers into and out of the rib waveguide. A photoluminescence lifetime of 138 μs and a pump absorption cross-section of  $1.79 \times 10^{-24} \text{ m}^{-2}$  were measured. A net internal gain of 3.75 dB/cm was achieved. Theoretical simulations predict that this gain could be further improved. Analysis of the results suggests that the difference between achieved and predicted gain is due to low quantum efficiency caused by two-ion cross-relaxation and up-conversion, as manifestly evident by yellow light emitted from the device. The molecular-level processes during the doped sol–gel fabrication that could lead to these phenomena are discussed, and strategies are suggested to overcome these limitations in order to approach the full potential gain.

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## 1. Introduction

Integrated optics biosensors are optical waveguide (WG) based devices capable of sensing of a specific bio-molecule analyte. The specificity of these sensors is achieved by fixation of “bio-receptors” such as antibodies to the sensor surface. Once the sensor is exposed to the sample liquid, the target bio-molecules are specifically bound to the bio-receptors on the sensor surface. The bound layer interacts with the evanescent fields of the guided waves and impacts their propagation coefficient. Channel waveguiding enables interferometric detection schemes such as WG-based Mach–Zehnder interferometers (WMZI). Although WMZI biosensors are most sensitive, their use is currently limited to optical lab environments due to their high cost and the need of delicate coupling of light source and detectors to the sensor chip restricts their use to optical lab environments. Recently, we reported the first pure monolithic solid state amplifier [1] realized in a Nd doped sol–gel rib WG. The amplifier was developed as part of an effort to establish a monolithic and integrated sol–gel-based light source for Lab-on-chip applications. The sol–gel method used is fully compatible with silicon technology. The sol–gel precursors are extremely inexpensive, the layers are applied by simple spin-coating on regular silica-on-silicon (SOS) and the fabrication is

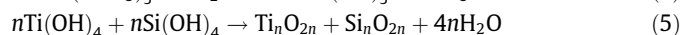
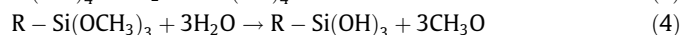
purely monolithic – no edge polishing or processing is required. In this paper, we present the sol–gel chemistry reaction system and processes used to realize this device and report on the phenomenon of strong up-conversion observed in the device. In addition, we present and analyze the comparison of the measured gain with that predicted by theoretical simulations of the device. The theoretical simulations indicate that the gain can be much improved relative to that measured by using a different host composition. The possible mechanisms responsible for that difference as well as the molecular-level processes of the doped sol–gel components that could lead to this phenomenon during its fabrication are analyzed. We conclude by suggesting different strategies regarding the sol–gel composition and preparation process for avoiding these limitations and for achieving the full potential gain predicted by the theoretical simulations.

## 2. Experimental

### 2.1. Nd doped silica–titania sol–gel reaction system and fabrication process

The sol–gel solution was prepared by reflux at 77 °C of the following precursors; γ-glycidoxypolytrimethoxysilane (GLYMO), titanium ethoxide, acetic acid and ethanol. The titanium-alkoxide precursor is added to reach a higher refractive index for the sol–gel guiding layer relative to that of the silica of the SOS substrate. The chemical reaction system is the following:

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Polymers formed with low water content are better suited for layer formation, using either spin-coating or dip coating [1,2]. Low water concentration prevents  $\text{TiO}_2$  dissolution and aggregation of large  $\text{TiO}_2$  colloids that cause optical scattering [3,4]. It also prevents uncontrolled gelling. On the other hand, in order to form a cross-linked  $\text{SiO}_2$ – $\text{TiO}_2$  siloxen network in a 3D structure, 2 moles of water are needed per mole of metal. A smaller molar fraction of water leads to chain polymer formation. Therefore the water content should be tightly balanced and controlled during the sol-gel process. For this reason, water is excluded from the precursors. The water required for the hydrolysis of titanium-ethoxide and GLYMO Eqs. (3) and (4), is formed in the esterification of the acetic acid and the ethanol Eq. (1) [5]. Once the water is formed, it is immediately consumed in the hydrolysis reactions and does not reach a concentration that would cause gelling and dissolution of titania colloids. The hydrolysis rate of titanium-ethoxide is also regulated by the availability of titanium-ethoxide, which is lowered by reacting with the acetic acid to form the titanium-acetate complex, and therefore suppresses the formation of the titania colloids. After 2 h of reflux, neodymium-nitrate-hexahydrate and aluminum-nitrate-nonahydrate were dissolved in ethanol and mixed with the solution. The guiding layer was fabricated by 4 cycles of spin-coating at 2000 rpm on a  $25 \times 25$  mm SOS substrate, followed by curing at  $100^\circ\text{C}$  on a hot plate. The chip was then annealed at  $800^\circ\text{C}$  for 1 h to remove residual organic groups and solvents, thus achieving the Nd–Al co-doped sol-gel silica-titania network with a molar ratio  $50\text{SiO}_2:50\text{TiO}_2:10\text{AlO}_{1.5}:1\text{NdO}_{1.5}$ .

## 2.2. Device structure and design

The channel WG has a rib cross-section. The rib (local) width is defined by the lateral distance between two 35 nm deep trenches etched into the guiding layer (Fig. 1). The rib WG starts with a  $50\text{ }\mu\text{m}$  wide section that contains an input grating and tapers linearly within 7 mm to a width of  $6\text{ }\mu\text{m}$ . This linear tapering is twice as long as the minimal length for adiabatic transition for the pump and signal modal fields [6]. The  $6\text{ }\mu\text{m}$  wide section is 3 mm long, and includes an output grating at its end. The grating couplers were calculated and designed using an accurate perturbation anal-

ysis [7]. To achieve a single diffraction order (i.e., one order into the WG and an unavoidable one into the substrate), the input angle of incidence for the pump ( $\lambda = 800\text{ nm}$ ) was designed to be  $-10^\circ$ , which implies a grating period of  $450\text{ nm}$  and an input angle of  $-53.4^\circ$  for the signal ( $\lambda = 1064\text{ nm}$ ). The grating depth was designed to be  $120\text{ nm}$  to achieve a coupling interaction length that corresponds to a waist of  $76\text{ }\mu\text{m}$  for the pump beam, which also suits well the mode lateral profile of the  $50\text{ }\mu\text{m}$  wide WG. The output grating period was designed to be  $470\text{ nm}$  to achieve an output angle of  $-45^\circ$ . The input and output grating designed lengths were  $157.5\text{ }\mu\text{m}$  and  $150.4\text{ }\mu\text{m}$ , respectively, to fairly cover the coupling interaction lengths.

## 2.3. Device fabrication process

The rib WG was defined by ZEP-mediated electron beam lithography (Raith 150) followed by  $\text{CHF}_3$  Reactive Ion Etching (RIE) of two  $9\text{ }\mu\text{m}$  wide trenches with  $35\text{ }\mu\text{m}$  depth. After the WG fabrication, a second lithography and etching process was performed to fabricate the gratings. An additional set of three un-tapered  $50\text{ }\mu\text{m}$ -wide devices was fabricated with identical input/output gratings but different lengths: 1 mm, 5 mm and 10 mm. This set was used for attenuation measurements. A similar set was fabricated for pump absorption measurements with an output grating period of  $346\text{ nm}$  designed to couple the pump to  $-45^\circ$ . A tapered WG with such an output grating was fabricated as well.

## 2.4. Measurement methods and setups

The sol-gel layer was measured using a spectroscopic ellipsometer (Wollam M2000DUV). The topography of the gratings and the rib was measured in an atomic force microscope – AFM (Di3000 Veeco). A tunable Ti-Sa laser (Coherent 890) was used for pumping and an Nd:YVO laser ( $\lambda = 1064\text{ nm}$ ) was used as the signal source. The pump and signal beams were focused on the input grating (from two different angles) by  $50\text{ mm}$  focal length lenses. The output signal was high-pass filtered (Schott RG 1000) to block the pump light ( $-41\text{ dB}$  at  $\lambda = 803\text{ nm}$ ) and measured with a powermeter (Nova Ophir). For attenuation measurements, the signal coupling into the different WGs was optimized in terms of maximizing the power emanating from the output gratings by fine-positioning of the input beam waist on the input grating and by scanning the input angles in the region of  $-53.4^\circ$ . Lifetime measurements were performed on a separate setup using an external acousto-optic modulator (NEC OD-88134) and a detector backed with a self-made trans-impedance amplifier connected to a digital scope (Tektronix TDS 2014). The modulated pump beam was prism-coupled to the planar WG and the detector was attached to the WG via the high-pass filter (Schott RG 1000) to suppress the pump light.

## 3. Results and discussion

### 3.1. Characterization of geometry and index

The refractive index of the sol-gel film was measured to be  $1.814$  at  $\lambda = 808\text{ nm}$  and evaluated to be  $1.803$  at  $\lambda = 1064\text{ nm}$ . Its thickness was  $360\text{ nm}$ . The index at  $\lambda = 1064\text{ nm}$  was extrapolated using the Cauchy formula fitted to the data measured at 400 wavelength points, since the measurement was limited to  $\lambda = 990\text{ nm}$  due to the ellipsometer. Fig. 2 shows an AFM topographic scan of the output grating at the end of the  $6\text{ }\mu\text{m}$  wide section of the rib WG. The grating teeth can be seen on the  $6\text{ }\mu\text{m}$  wide rib WG located between the two  $9\text{ }\mu\text{m}$  wide trenches. The grating teeth can also be seen at the edge of the AFM scan on the left, as they were written and etched few microns beyond the rib WG trenches.

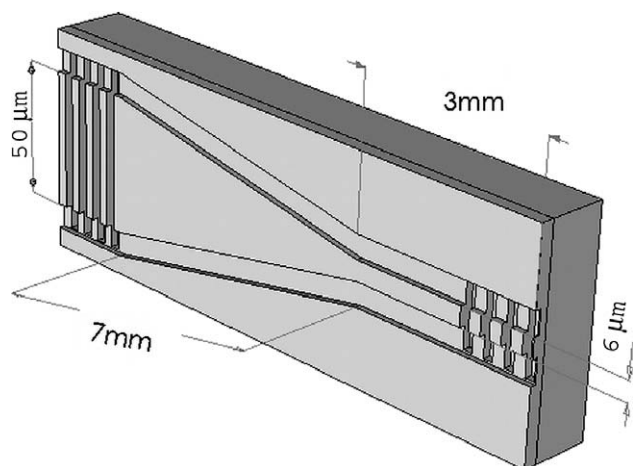


Fig. 1. Amplifier 3D scheme.

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