oA4-1

FABRICATION AND CHARACTERIZATION OF ORGANIC-INORGANIC OPTICAL WAVEGUIDES ON SILICON BY SOL-GEL TECHNOLOGY

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ABSTRACT: We report on the fabrication and characterization of waveguide thin films on silicon by ultraviolet light imprinting in a new photosensitive, organically modified aluminophosphate sol-gel silica glass prepared by a one-step dip-coating process. Appropriate combinations of ultraviolet light exposure time, sol-gel film thickness and postbake parameters produce waveguides suitable for optical telecommunication applications.

KEYWORDS: waveguides, sol-gel glass, silicon, ultraviolet, telecommunication

1. Introduction

We have developed a process to produce high quality sol-gel silica glass thin films for integrated optics waveguide fabrication [1]. This process is appealing not only for its simplicity, but also because low temperature wet-processing is largely compatible with generic, planar on silicon and III-V device benches. We have used this process to fabricate waveguides on silicon [2, 3, 4, 5]. The waveguides are made by a simple ultraviolet (UV) light imprinting in a hybrid sol-gel silica glass on silicon substrate. The waveguides are achieved in a few steps using inexpensive equipment. In the present work, we have developed a new low refractive index aluminophosphate sol-gel for integrated optics applications. UV-light imprinting was used to make slab waveguides in the new sol-gel. An in-depth study of the optical properties of the sol-gel thin films (slab waveguides) on silicon was carried out. In section 2, we explain the sol-gel material and thin film waveguide fabrication process. Section 3 reports on the sol-gel thin film characterization. Section 4 discusses the results.

2. Fabrication

The sol-gel was prepared according to the procedure outlined in references [1-4], with the following modification: The photosensitive silicon alkoxide precursor used to prepare the sol-gel glass film was methacryloxypropyl trimethoxysilane $H_2C=C(CH_3)CO_2(CH_2)3Si(OCH_3)_3$ (MAPTS), that contain organic reactive function methacryl ligand that may be polymerized further by ultraviolet light or finally heat treatment. Trimethyl phosphate $P(O)(CH_3O)_3$ and isobutoxyaluninooxytriethoxysilane (the Al-O-Si alkoxide) were used as the sources of Al^{3+} and P^{5+} in the glass. They were chosen as the dopants to modify the refractive index of the material and the thickness of the glass films. MAPTS was prehydrolyzed during two hours with aqueous solution of hydrochloric acid (10-2 M) as a catalyst. The aluminum and phosphorous compounds were added and stirred together. Water was added and reactions were allowed to continue for approximately fifteen hours after adding the water. Finally, a

TOUAM T. et al – Oral A4-1

photoinitiator (IRDATURE 184, CIBA) was added. The mixture was passed through a $0.2 \,\mu m$ filter to obtain a free-flowing sol.

The sol-gel thin film (slab waveguide) fabrication process consisted of four steps: gel preparation, dip-coating, UV-light exposure and postbake. Figure 1 summarizes the sol-gel glass waveguide fabrication process.



Figure 1: Thin film waveguide fabrications flow-chart.

The substrates were cleaned with detergent, rinsed in flowing deionized water and soaked for 10 min. in warm trichloroethylene. Rinsing with acetone and isopropanol, and drying with nitrogen completed the procedure. Thin films were deposited at different drawing speeds on silicon substrates having a 2 μ m thermally grown SiO₂ buffer layer. Films were then immediately prebaked at 100 °C in air for 60 min. to stabilize them prior to UV-light exposure. In addition, the heating step hardens the films sufficiently to prevent them from adhering to the mask during photo imprinting. An Oriel deep UV-light ($\lambda = 220-320$ nm) illumination system was employed to polymerize the vinyl substituents. This illumination system is designed to produce a collimated and uniform beam of deep UV-light. This new light source is different from the one used in our previous works. The effect of the new light source will be seen later.

Exposure of the sol-gel to UV-light sensitizes the vinyl monomer substituent to polymerization, and the refractive index of the regions of the film exposed to UV-light radiation increases relative to the unexposed regions. The UV-light penetrated into the sol-gel glass down to the silica buffer layer. Finally, a postbake step at for 1 hour was used to harden the film and further enhance the refractive index in the photo-defined regions. A postbake

temperature of 130 °C was usually used. However, the effect of the postbake on the film characteristics was also studied between 130 °C to 160 °C.

3. Characterization

The deposited unexposed sol-gel thin films exhibited higher refractive index than the silica buffer layers allowing guided modes to be excited in the slab waveguides. A Sloan DekTak auto-leveling profilometer and a prism coupling method [6] with a 0.6328 μ m HeNe laser were used to determine the thickness and the refractive index of the films. Figure 2 summarizes the results. It shows an excellent agreement between the DekTak and prism coupling measurements. The results confirm that UV-light penetrates into the sol-gel glass and reaches the silica buffer layer. This is different from previous process [7, 8, 9] where the UV-light was confined to a finite penetration depth of ~ 3.5 μ m in the sol-gel thin film. The deeper penetration of the UV-light in the sol-gel is due to more transparent new sol-gel material and the new UV-light source that has relatively high intensity deep UV-light emission. We have used a step refractive index function for both unexposed and exposed films in prism coupling characterization.



Figure 2: Variation of sol-gel glass film thickness with drawing speed. The films were post baked at 130 °C for 60 min.

Prior to UV-light exposure, the refractive index of the sol-gel glass film was measured to be 1.489 and 1.487 for TE and TM polarization, respectively. After the exposure to the UV-light, sol-gel thin film refractive index increased by Δn , reaching its maximum value after 45 min. Beyond this time, Δn plateaus at about 0.008 compared to the unexposed film. Figure 3 summarizes the variation of Δn with UV-exposure time.

TOUAM T. et al – Oral A4-1



Figure 3: Refractive index increase, Δn , in sol-gel films as a function of UV-light exposure time.

The effect of the sol age on the thickness of the film was also studied. A container of sol-gel was prepared and thin films were made periodically during two weeks. The container was sealed and stored in dark between successive dip-coatings. It was unsealed only for a short time to dip-coat a few samples. Figure 4 depicts the variation of the sol-gel thickness with sol age for different drawing speeds. The results indicate that there is a slight increase in the prepared sol-gel thin film thickness. This may be due to the solvent evaporation during the time that the sol-gel in the container was exposed to air, resulting in an increase in the viscosity of the sol-gel.

Additional experiments were performed to study the effect of light and temperature on exposed and unexposed sol-gel thin films. They were left in the laboratory exposed to room light for a period of 30 days. During this period, the propagation constant of the guided modes in these waveguides were periodically measured. There was not any measurable change in the optical properties of the exposed and unexposed films. The effect of intense UV-light on the exposed and unexposed films was investigated by the following experiment:

- Thin films were prepared on silicon as explained above.

- The samples were prebaked at 100 °C for 60 min.
- The samples were postbaked at temperatures ranging from 130 °C to 160 °C for 60 min.

- The samples were exposed again to UV-light for 60 min.

Figure 5 summarizes the results. The refractive index of the sol-gel thin film increases up to 0.005.

4. Discussion

We have developed a new aluminophosphate sol-gel material for integrated optics device fabrication. It was shown that the film thickness and refractive index are reproducible, which makes this material and process suitable for device manufacturing. The results indicate that there is not a significant variation in film thickness with sol age. As for the refractive index increase, Δn remains unchanged for the same UV-exposure time, regardless of sol age.

There is not any change in the refractive index of sol-gel films after postbake if they are exposed to room light. However, the results of Figure 5 indicate that if the unexposed sol-gel undergoes exposure to intense UV-light, its refractive index increases. This increase depends on the postbake temperature. There are two components to the refractive index increase of these films in the 130 °C -160 °C postbake range: 0.003 due to UV-light exposure, ~0.00007 / °C due to temperature. It is expected that the UV-light exposed film undergoes similar changes. The experiments are presently being carried out.

The fabricated sol-gel thin films are birefringent with $n_{TE} - n_{TM} = 0.002$. Our preliminary experiments indicate that it may be possible to eliminate the birefringence in the waveguides by additional UV-light exposure and/or postbake, and by suitable selection of postbake temperature.



Figure 4: Film thickness variations with sol age. V = drawing speed.

We are using the new process to manufacture power splitters and WDMs. Low-loss high performance devices are feasible.



Figure 5: Refractive index increase, Δn, in sol-gel films as a function of postbake temperature. The films were exposed to UV-light for 60 min.

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TOUAM T. et al - Oral A4-1

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