FABRICATION OF LOW-LOSS CHANNEL WAVEGUIDES IN SOL-GEL GLASS ON SILICON SUBSTRATES

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A new, low-cost process for the fabrication of silica-on-silicon integrated optic components is described. Thick (> 10 μm) films of P₂O₅ doped silica are deposited as multilayers by spin-coating, with each layer being consolidated by rapid thermal annealing before application of the next. By control of the process parameters, this method can produce optical quality films with low stress. These films are patterned by photolithography and reactive ion etching to form ridge waveguides. The guides are then reflowed by rapid thermal annealing, and buried under further layers of sol-gel glass. In this paper, the essential features of the fabrication process are discussed, and results are presented for optical losses.

1. INTRODUCTION

We have devised a sol-gel process for the deposition of a passive optical material, which can be used to fabricate channel waveguide devices. The material is SiO₂ doped with a variable proportion of P₂O₅. The P₂O₅ dopant alters the refractive index of the silica, allowing the construction of waveguide devices based on multilayer structures containing different P₂O₅ concentrations. It also lowers the melting point of the glass, allowing a controlled reflow step to be used to reduce sidewall scattering losses in passive devices.

As with our previous TiO₂ : SiO₂ process¹,², the material is prepared from a sol-gel precursor, whose primary ingredient is Si [OC₂H₅]₄ (TEOS, or tetraethylorthosilicate). This is spin coated onto Si wafers and densified by rapid thermal annealing. Thick films are built up by iteration of the process cycle spin/densify as before. However, the incorporation of the P₂O₅ dopant alters the conditions for rapid thermal annealing³ and for any subsequent thermal processing steps.

2. PREPARATION OF SOL-GEL PRECURSORS

Sol-gel precursors are prepared using a two-step hydrolysis, as shown in Figure...
In the first step (see Figure 1a), a partially hydrolysed solution of Si [OC₂H₅]₄ known as Solution A is formed. Here, the aim is to replace on average one OC₂H₅ group in each TEOS molecule by an OH group (a state referred to as R = 1). The hydrolysis is performed by adding an appropriate quantity of water and acid catalyst to a solution of Si [OC₂H₅]₄ in alcohol, and then refluxing. Both ethanol and 2-propanol have been tried as solvents, and found to give similar results. The acid and water are introduced together as a 0.1 M aqueous solution of HCl (Solution H).

In the second step (see Figure 1b), the P₂O₅ dopant is incorporated, and the mixture is further hydrolysed (to R = 2) to give the final sol, which is referred to as Solution A/P. The P₂O₅ dopant is introduced as a 1 M solution of P₂O₅ in alcohol (Solution P). This is added to an appropriate quantity of previously prepared Solution A. Then further quantities of acid catalyst and water are added, and the mixture is refluxed a second time.

**FIGURE 1**
Preparation of sol-gel precursors: a) 1st stage, and b) 2nd stage hydrolysis.

3. SPIN-COATING AND RAPID THERMAL ANNEALING

Prior to spin-coating, the sol may be further diluted with solvent to reduce its viscosity, depending on the desired application. For example, a dilution of 1 part solvent to 1 part sol (referred to as 1 : 1 dilution) is used for buffer and guide layer deposition, while 2 : 1 dilution is used for guide burial. The diluted sol is dispensed onto a silicon wafer from a syringe fitted with a 0.1 μm in-line filter. Spin-coating is
then performed for a fixed time (40 secs) at a fixed rotational speed in the range 1000 rpm - 5000 rpm. Again, the speed used depends on the application; low speed is used for planar layer deposition and high speed for guide burial.

In all sols tested, the single-layer film thickness \( t \) obtained at a spin speed \( \omega \) has followed (to good approximation) the law:

\[
t = k (\omega)^{-0.52}
\]

where \( k \) is constant for any given sol. This is illustrated by Figure 2, which shows the variation of single-layer thickness with spin speed for different PSG sols. In these data, the thickness ranges from 0.07 \( \mu m \) to 0.73 \( \mu m \), a considerable range. However, all variations are linear and have similar slopes when plotted on a log-log scale.

![Figure 2](image)

**FIGURE 2**

Variation of single-layer film thickness with spin speed for different PSG sols.

Two main factors determine the thickness obtained at a given spin speed (i.e. the value of the constant \( k \)). The first is the molar fraction of \( P_2O_5 \) contained in the sol. It is found that an increase in the \( P_2O_5 \) content results in a small increase in layer thickness. This may be explained, at least partially, by the fact that Solution P is markedly more viscous than Solution A. The second factor is the final dilution in alcohol. This has a considerable effect. For example, Figure 3 shows the variation of single-layer thickness with final dilution for films of ethanol-based sol-gel PSG (5 mol\% \( P_2O_5 \) content, spun at 2000 rpm for 40 seconds and annealed in oxygen at 950°C for 10 seconds). To a good approximation, the thickness varies linearly with the volume fraction of ethanol, from a maximum for undiluted sol (0 : 1) to a
minimum of zero for pure ethanol (\(\infty : 1\)). Dilution with equal parts of solvent (for example) more than halves the thickness of the deposited layer.

![Graph showing the variation of film thickness with volume fraction of ethanol for 5 mol% PSG.](image)

**FIGURE 3**

Variation of film thickness with volume fraction of ethanol for 5 mol% PSG.

Rapid thermal annealing (RTA) is performed in O\(_2\) for 10 seconds after each spin-coating step. As with our TiO\(_2\) : SiO\(_2\) deposition process, we have found that the RTA temperature must be higher than a particular value for crack-free deposition of thick multilayer films. In previous experiments, we have deduced that cracking occurs when the film stress is tensile at any point in the process. By measuring the wafer curvature at room temperature, we estimated the stress at the annealing temperature, and hence adjusted the RTA temperature to obtain either zero or a slightly compressive stress during annealing. This allowed us to build up thick (5 - 10 \(\mu\)m) films with a \(\text{P}_2\text{O}_5\) content of 0 - 15 mol%.

Figure 4 shows the variation of the optimal rapid thermal annealing temperature with \(\text{P}_2\text{O}_5\) content for sol-gel PSG spun at 2000 rpm for 40 seconds and annealed in O\(_2\). As can be seen, an increase in the \(\text{P}_2\text{O}_5\) content reduces the RTA temperature quite dramatically, by roughly 20°C per mol%. This is beneficial, because thick films may be built up without introducing the high degree of wafer damage observed in our TiO\(_2\) : SiO\(_2\) process (for which the annealing temperature was 1075°C).

An increase in the \(\text{P}_2\text{O}_5\) content has an additional, less desirable side-effect. When films with a high \(\text{P}_2\text{O}_5\) content are annealed at too high a temperature, they become grey and opaque (presumably due to phosphorous out-diffusion). For 15 mol% PSG this occurs at 900°C, while for 25 mol% PSG it occurs at annealing temperatures as low as 650°C.
FIGURE 4
Variation of annealing temperature, reflow temperature and film refractive index with P₂O₅ content for sol-gel PSG.

Film refractive index \( n \) has been found to vary approximately linearly with P₂O₅ content, as expected. For example, Figure 4 also shows refractive index at 633 nm versus P₂O₅ content for films coated at 2000 rpm and annealed in O₂ at 950°C for 10 seconds. The variation is described approximately by:

\[
n = 1.457 + 0.001 \, y
\]

where \( y \) is the P₂O₅ content in mol\%. The value at \( y = 0 \) is slightly lower than the value for pure silica, indicating that the films may be incompletely densified.

4. CHANNEL WAVEGUIDE FABRICATION

The process for channel guide fabrication is directly analogous to that previously developed for the TiO₂ : SiO₂ material system\(^1,2\). A planar bilayer structure is first formed, using a layer of higher index material (the core layer) above a layer of lower index material (buffer layer). Typically, we have used a 5 μm thick layer of 10 mol% P₂O₅ : SiO₂ for the core and a 10 μm thick layer of 5 mol% P₂O₅ : SiO₂ for the buffer, to obtain a \( \Delta n \) of \( \approx 5 \times 10^{-3} \) between core and cladding.

The bilayer is masked with Cr and the core layer reactive ion etched to yield guide cores of rectangular cross-section. After etching, the cores are reflowed at a high temperature to transform the guide cross-section to a circular form and remove sidewall roughness introduced by the RIE step. Here, an important difference from
our previous process is that the P2O5 dopant significantly reduces the glass melting temperature. The reflow temperature is therefore lower, and there is scope for process modification to obtain different core cross-sectional shapes. After reflow, the cores are buried by further spin coating and rapid thermal annealing. This burial process can lead to a high degree of planarisation if carried out correctly.

Reactive ion etching is performed in an Oxford Plasma Technology RIE 80 using a 3-gas process (Gas flows: 25 sccm CHF3, 25 sccm Ar, 1 sccm O2; electrical conditions: 140 W RF power, 420 V DC bias; pressure: 50 mTorr). O2 is included to eliminate micromasking. Under these conditions, the most common PSG compositions tested (5 and 10 mol% P2O5 : SiO2) both gave etch rates of \( \approx 500 \) Å/min. This is comparable to the rate obtained with TiO2 : SiO2. However, there is a significant reduction in the degree of anisotropy obtained, so that final guide widths are reduced from their nominal values by 1.5 - 2 μm. This is a considerable variation, which must be compensated for if guides of appropriate width are to be obtained. Figure 5a shows a guide core formed from 10 mol% P2O5 : SiO2, immediately after etching. Lateral erosion of the sidewalls may clearly be seen.

![Figure 5](image_url)

**FIGURE 5**

Guide core formed in 10 mol% P2O5 : SiO2, after a) RIE, and b) reflow.

Two reflow methods have been investigated: heating for a short time at a high temperature in a rapid thermal annealer, and heating for longer times at a lower temperature in a tube furnace. In both cases, full reflow can easily be obtained. There is some evidence to suggest that, in addition to being more convenient, rapid reflow is beneficial in reducing surface roughness caused by phosphorus out-diffusion. However, this is inconclusive at present.

As mentioned earlier, the reflow temperature depends strongly on the glass composition. Figure 4 shows the approximate variation with P2O5 content of the
temperature needed for full reflow in a 20 minute process. Over the range of composition considered, the variation in reflow temperature is \( \approx 175^\circ\text{C} \). For 10 mol\% \( \text{P}_2\text{O}_5 : \text{SiO}_2 \), the reflow temperature is \( \approx 140^\circ\text{C} \) below that required for the \( \text{TiO}_2 : \text{SiO}_2 \) process, a further advantageous reduction.

The reflowed channel waveguide cores are buried under material having the same composition as the buffer. In order to prevent cracking, a diluted sol (typically 2 : 1) is employed, and the spin speed is gradually decreased from an initially high value as the burial proceeds. Figure 6 shows an SEM view of the cross-section of a completed guide, which has been polished and then etched in buffered HF to highlight the core/cladding boundary. The HF etch step has also clearly defined the structure of the laminations formed during the burial process.

![FIGURE 6](image)

Cross-section of a completed channel guide in sol-gel phosphosilicate glass.

5. OPTICAL MEASUREMENTS

A number of chips containing straight waveguides of nominal width 4 - 7 \( \mu \text{m} \) were fabricated to demonstrate the use of the passive \( \text{P}_2\text{O}_5 : \text{SiO}_2 \) materials system. In each case, the buffer layer consisted of 10 \( \mu \text{m} \) of 5 mol\% \( \text{P}_2\text{O}_5 : \text{SiO}_2 \) and the core of 5 \( \mu \text{m} \) of 10 mol\% \( \text{P}_2\text{O}_5 : \text{SiO}_2 \) (height specified before reflow). The waveguides were assessed by measuring the fibre-device-fibre insertion loss at 1525 nm wavelength. For example, Figure 7 shows average insertion losses for 3 cm guide length versus nominal guide width. The insertion losses are high, especially for the TM mode, but reduce significantly as the guide width increases.
FIGURE 7
Fibre-device-fibre losses at $\lambda = 1525$ nm for 30 mm lengths of passive channel waveguide fabricated in phosphosilicate sol-gel glass.

Propagation and coupling losses were measured separately for the devices above, by comparing fibre-device-fibre and fibre-device-multimode fibre insertion loss. For guides of 7 $\mu$m nominal width, fibre coupling losses were 0.3 dB per facet, and TE and TM propagation losses were 0.45 and 2.0 dB/cm, respectively.

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REFERENCES