Rare-Earth-Ion-Doped Al_2O_3 Waveguides for Active Integrated Optical Devices

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ABSTRACT

Reactively co-sputtered amorphous Al_2O_3 waveguide layers with low propagation losses have been deposited. In order to define channel waveguides in such Al_2O_3 films, the etching behaviour of Al_2O_3 has been investigated using an inductively coupled reactive ion etch system. The etch rate of Al_2O_3 and possible mask materials was studied by applying various common process gases and combinations of these gases, including CF_4/O_2, BCl_3, BCl_3/HBr and Cl_2. Based on a comparison of the etch rates and patterning feasibility of the different mask materials, a BCl_3/HBr plasma and and standard resist mask were used to fabricate channel waveguide structures. The etched structures exhibit straight sidewalls with minimal roughness and etch depths of up to 530 nm, sufficient for defining waveguides with strong optical confinement and low bending losses. Low additional propagation losses were measured in single-mode Al_2O_3 ridge waveguides defined using the developed etch process. In initial investigations, Al_2O_3:Er layers fabricated using the same deposition method applied for the undoped layers show typical emission cross-sections, low green upconversion luminescence and lifetimes up to 7 ms.

INTRODUCTION

Rare-earth-ion-doped aluminum oxide layers are promising for active integrated optics applications, such as waveguide amplifiers and lasers [1]. Due to this potential, the deposition of high-optical-quality rare-earth-ion-doped films of Al_2O_3 have been the subject of intense study [2][3][4]. Recently, losses as low as 0.11 dB/cm at 1522 nm [5] have been measured in undoped Al_2O_3 layers, demonstrating the potential for low intrinsic waveguide losses, which is a necessary condition for sufficient net gain in the doped layers. In order to realize high-quality integrated active waveguide devices in such low-loss Al_2O_3 layers, a reliable patterning technique is required. For Al_2O_3:Er, Ar-ion beam milling (or sputtering) has previously been used to define ridge waveguide structures [6], and recently wet chemical etching has also been employed [7]. However, both techniques limit both the overall resolution of the process and the steepness of the sidewall profile. Furthermore, the etch depths were limited to 300 nm in the Ar milling case and less than 400 nm for wet etching of Al_2O_3.

With the aim of achieving sufficient gain in active waveguide devices, a fabrication technique is required with high resolution, sufficient etch depth and low additional losses introduced by the etch process itself. For high resolution, good selectivity to the mask material and steep (anisotropically etched) sidewalls are required. Deeply-etched channels (as opposed to shallow-etched ridge-type structures) also may be required for high confinement to achieve good overlap of the pump and signal beam and minimize the bend radius (without significantly adding to the losses). Finally, for low additional losses due to channel etching, smooth sidewalls are required. The plasma etching characteristics of Al_2O_3 films in different chemistries have been widely studied for various applications [8][9][10][11]. RIE of optical waveguides in Al_2O_3 films has been reported [12], but the process involved a complicated 3-level masking procedure and

utilized a metal Cr-mask, which is less desirable than other materials, because metals can introduce extra losses in the waveguide. In this paper, the etching behaviour of amorphous Al_2O_3 films and possible masking materials are investigated using an inductively coupled plasma (ICP) RIE system. Based on the etching data, an optimized process has been developed for fabricating high-quality, low-loss channel waveguides. With the future aim being the application of this fabrication process to optical waveguides and devices with low background losses in rare-earth-ion-doped Al_2O_3 layers, initial promising results regarding co-sputtered Er-doped Al_2O_3 layers are also presented.

EXPERIMENTAL

The Al_2O_3 layer deposition was optimized by applying a reactive co-sputtering system, which has been discussed in detail elsewhere [2][5]. Doped and undoped Al_2O_3 layers with ±0.8% thickness non-uniformity over 50x50 mm^2 were grown from high-purity Al and Er targets on 8-µm thermally oxidized 100-mm Si substrates. The refractive index was 1.659 at 633 nm. The optical loss of the layers, ranging in thickness from approximately 500 nm to 800 nm, decreased with increasing deposition temperature, as shown in Figure 1. Optical loss as low as 0.11 dB/cm at 1523 nm was demonstrated at 550°C deposition temperature.

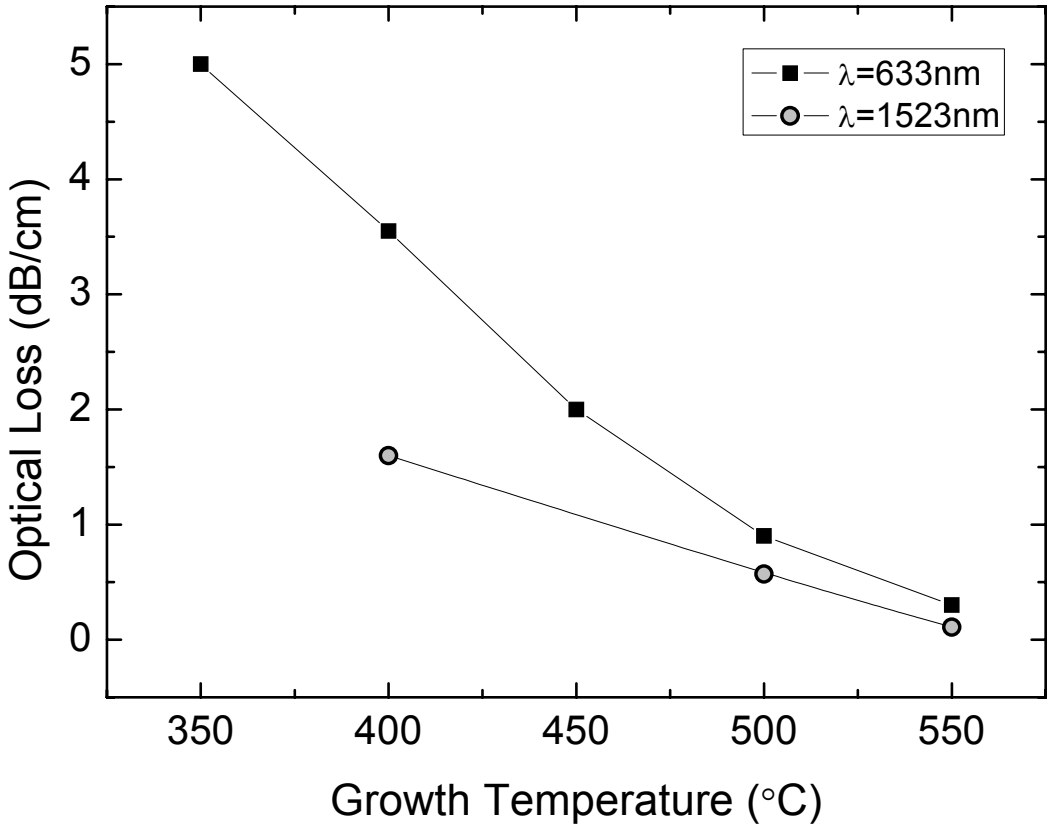

Figure 1: Optical loss of as-deposited Al_2O_3 layers as a function of deposition temperature, measured at wavelengths of 633 nm and 1523 nm.

The etch experiments were carried out using an Oxford Plasmalab 100 inductively-coupled plasma (ICP) RIE system. The system was designed for 100-mm wafers, which were introduced to the chamber through a load-lock and fixed on a substrate holder with water-cooled electrode. The ICP source was controlled by a 3-kW, 13.56-MHz RF generator, while substrate bias was controlled separately by a 600-W, 13.56-MHz RF generator. Various standard process gases and combinations of these gases were used, including BCl_3, BCl_3/HBr (50%:50%), CF_4/O_2 (90%:10%) and Cl_2. The total gas flow was held constant at 50 sccm (measured by mass flow control units), while process pressure (measured by a capacitance manometer gauge) was maintained as low as possible, varying between 7-12 mTorr. Unless otherwise stated, the ICP power was held constant at 1500 W and the applied RF electrode power was varied from 100 to 400 W. In addition to the Al_2O_3 films discussed, various potential common mask materials were also investigated in terms of patterning methods, etch selectivity and possible removal after etching. Accordingly, 3-µm-thick plasma-enhanced chemical vapour deposited (PECVD) SiO_2 and Si_3N_4 films, standard 1.5-µm photoresist films, and 200-nm-thick electron-beam evaporated Ni and Cr layers were also prepared on Si substrates.The etch rates of the films were determined by measuring the film thickness before and after the etch process using a spectroscopic ellipsometer, while the etch rates of the silicon substrates and Ni and Cr layers, patterned prior to etching by photolithography and wet chemical etching, were measured using a Dektak surface profilometer. The channel waveguide fabrication process is discussed in the following section.

RESULTS

A) ETCHING OF Al_2O_3 AND MASK MATERIALS

The etch rate of the Al_2O_3 films was investigated as a function of applied RF power for various plasma compositions. Figure 2 shows the measured etch rate of Al_2O_3 films as a function of RF power for CF_4/O_2 (90%:10%), BCl_3 (100%), BCl_3/HBr (50%:50%) and Cl_2 (100%) gases. The highest etch rate, 257 nm/min was measured for CF_4/O_2 at 400 W and the etching in CF_4/O_2 is the most strongly dependent on RF power, indicating that etching under these conditions also depends strongly on the energy of ions directed onto the substrate (it is strongly ion-assisted). The potential F-based etch products are expected to have lower volatilities (based on higher melting and boiling points) than Cl-based products, which is consistent with the observed stronger effect of ion energy on removing material from the surface and assisting the reaction. These data generally agree with the previous etching results for similar Al_2O_3 layers in CF_x gases [8]. The etching in Cl-containing gases is shown to be less dependent on RF-power, indicating that chemical etching, rather than ion-assisted etching, more strongly affects the etch rate and behaviour. Of the Cl-containing gases, the etch rate is shown to be significantly higher in BCl_3 than Cl_2 or BCl_3/HBr (50%:50%).

The etch rates of several common mask materials, chosen for structuring Al_2O_3 mainly because these mask materials can be easily deposited and patterned in our laboratory, were also measured. The principle requirements for a good mask material are that it can be easily patterned, it has a high selectivity compared to the material to be etched (for good pattern transfer and high resolution), and it must be possible to selectively remove the mask material from the substrate after etching. Generally, selectivities much greater than 1 are preferred, however for more highly stable dielectric materials such as Al_2O_3 we expect lower selectivites. Table 1 shows the maximum selectivities measured for Al_2O_3 to the various prospective mask materials. The process window used to determine the mask material etch rates was essentially the same as that used for the Al_2O_3 etching experiments (ICP power = 1500 W, RF Electrode Power = 100 to 400 W, pressure = 6 to 14 mTorr, total gas flow = 50 sccm). The selectivity in general was found to be 2 times lower for patterned as opposed to unpatterned mask layers.

For all mask materials except Ni, the selectivity versus Al_2O_3 was generally higher in BCl_3 and BCl_3/HBr. The selectivities for Si_3N_4 were > 1 for these gases, however selective removal of Si_3N_4 from the Al_2O_3 layer after etching was found to be a problem. The selectivities to Ni are also quite high in comparison to other materials, as we would

expect for a harder metal mask, however, if possible it is better to avoid metal masks for optical applications, as any residual metal remaining after etching and the mask removal can introduce additional optical losses. For Cr, significant data is missing because it was realized early in the experiments that patterning mask layers with smooth sidewalls was difficult – perhaps due to the layer deposition process or quality of the Cr films. Both metal layers (Ni and Cr) were found to react with the Cl-containing gases leaving residue on the surface. Therefore, for reasons of simplicity, it was decided to use photoresist as a mask, varying the parameters to improve the selectivity and using the BCl_3/HBr system because this gave the highest overall selectivities.

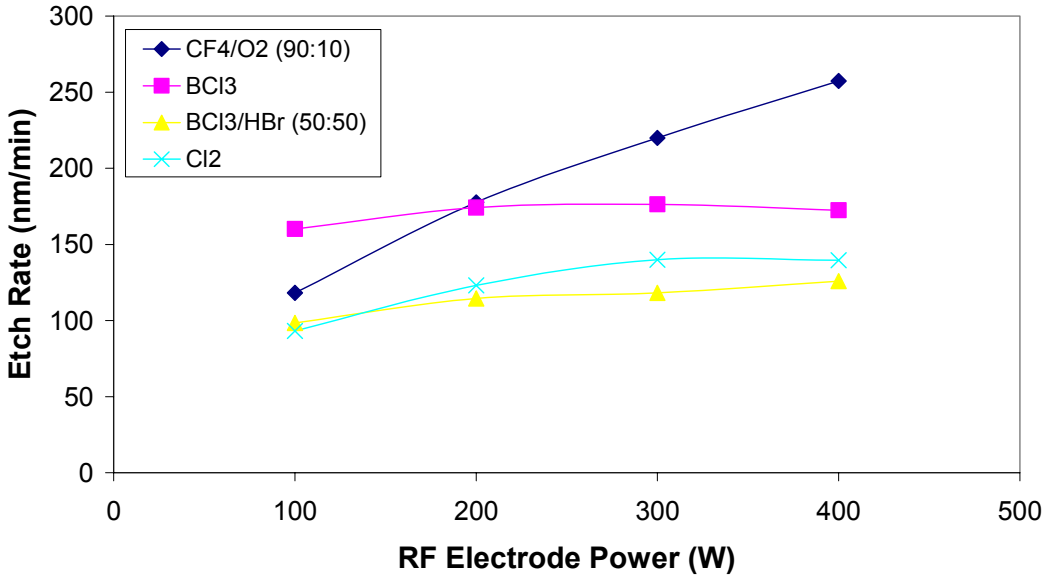

Figure 2: Etch rate as a function of RF power for Al_2O_3 films in CF_4:O_2 (90%:10%), BCl_3 (100%), BCl_3:HBr (50%:50%) and Cl_2 (100%) at a total flow rate of 50 sccm.

Table 1: Maximum selectivities of Al_2O_3 to possible mask materials, for various process chemistries, at a total gas flow rate of 50 sccm, ICP power of 1500 W, varying RF electrode power (100 to 400 W) and process pressure in the range of 6 to 14 mTorr.

	Process Gas			
Material	CF_4/O_2 (90%:10%)	BCl_3	BCl_3/HBr (50%:50%)	Cl_2
Photoresist	< 0.20	0.67	0.54	0.16
PECVD SiO_2	0.60	0.95	0.80	0.70
PECVD Si_3N_4	0.35	1.35	1.80	0.65
Silicon	0.39	0.98	2.15	0.30
Nickel	5.82	-	3.51	1.38
Chromium	-	-	-	< 0.94

B) CHANNEL WAVEGUIDE FABRICATION

Al_2O_3 channel waveguides ranging from 1.2 to 8.0 µm in width were fabricated using a BCl_3/HBr gas mixture and 1.5-µm photoresist mask layer patterned by standard lithography. These process gases have previously been demonstrated as a suitable gas mixture for etching straight, vertical ridge structures with smooth sidewalls in sapphire (crystalline Al_2O_3) [13][14], with channel waveguides being demonstrated in Ti:sapphire layers using a BCl_3/Cl_2 mixture [15] (similar structures were also obtained by Ar-ion beam milling [16] and have later been demonstrated to lase [17]). The optimized gas ratio in terms of selectivity for the amorphous Al_2O_3 films was found to be 5:2 (BCl_3:HBr) and optimized process parameters were ICP power = 1750 W, RF electrode power = 25 W, pressure =12 mTorr and a total gas flow of 35 sccm. Figure 3 shows the channel waveguide etching process. In Figure 3 (a) and (b) the resist mask on top of the Al_2O_3 layer is shown before and after etching, respectively. Figure 3 (c) and (d) show the resulting Al_2O_3 channel structures, (etched to a depth of 340 nm in this case).

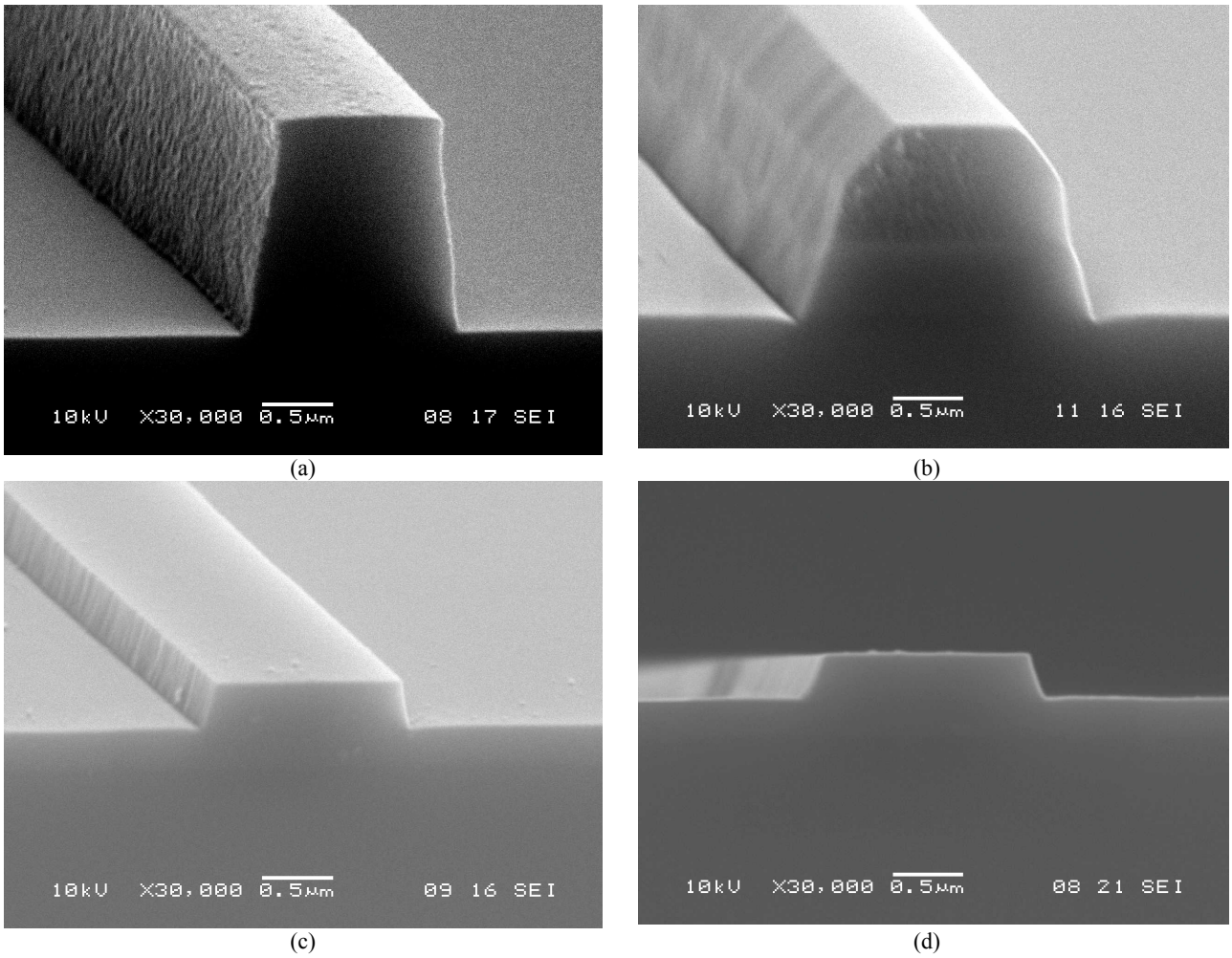

Figure 3. SEM micrographs of (a) an Al_2O_3 layer with 1.7 µm-thick photoresist mask on top before etching; (b) the etched Al_2O_3 layer with resist mask still on top after applying a BCl_3/HBr plasma; and (c) and (b), resulting Al_2O_3 channel waveguide structures.

The etch rate was measured to be 59 nm/min and the selectivity to the resist mask was 0.76 (which is higher than the 0.54 in Table 1 because of lower applied RF electrode power), allowing maximum etch depths of > 500 nm. The channel sidewall angle ranged from 56º to 68º depending on the waveguide width and a slight trench was observed beside the waveguide, typical of RIE processes. As the SEM pictures illustrate, sidewall roughness in the channels may be attributed to roughness in the initial photoresist mask layer.

In order to test the additional losses introduced by channel etching, single-mode ridge waveguide structures were patterned in low-loss Al_2O_3 layers. Waveguides were defined using the optimized recipe, to an etch depth of 230 nm in a 690-nm-thick film. The etch-depth uniformity over the substrate was found to be ± 5%. Prior to etching, the optical losses of the film were measured to be 0.57 ± 0.06 dB/cm at a wavelength of 1523 nm using the prism coupling method. The optical losses of 1.5- and 2.0-μm-wide ridge waveguides, designed to be single mode at wavelengths around 1550 nm, were investigated using a fiber butt-coupling setup and broadband erbium-doped fiber amplifier (1520-1580 nm) source. By taking into account reflection losses and losses due to mode mismatch at the input and output facet, propagation losses around 1 dB/cm were determined for both waveguide widths indicating that only rather small additional losses are introduced by the dry-etching process.

C) Er-DOPED LAYERS

Er doping of the Al_2O_3 layers was realizied by making use of the reactive co-sputtering system described in [3]. Layers with ±0.8% thickness non-uniformity over 50x50 mm^2 were grown from high-purity Al and Er targets on thermally oxidized <100> Si substrates.

The erbium concentration of the $Er:Al_2O_3$ layers was analyzed using Rutherford backscattering spectroscopy (RBS). This method is a powerful technique for the characterization of $Er:Al_2O_3$ layers, as it enables accurate determination of Er-concentration, simultaneously providing information on the depth profile. The RBS investigation of the layers showed that the Er-doping profile is uniform throughout the thickness of the film. Variation of the sputtering power on the Er-target of up to 25 W resulted in incorporation of Er in the layers with concentrations of up to approximately 4.5×10^{20} cm^{-3}. The refractive index of the films was found to steadily increase from 1.659 to about 1.673 at 633 nm, as the Er-target power was varied from 0 to 25 W. The absorption spectrum of the doped layers shows the typical absorption bands of Er^{3+}, with peaks at 1529 nm ($^4I_{13/2}$), 977 nm ($^4I_{11/2}$), 797 nm ($^4I_{9/2}$), 652 nm ($^4F_{9/2}$), and 522 nm ($^2H_{11/2}$). Lifetimes up to 7 ms were measured for the $^4I_{13/2}$ level in the doped films.

Finally, optical losses in the Er-doped layers remained low and waveguide propagation of 633-nm light over 9 cm was easily achieved. Pumping with 10 mW of 1480-nm pump light coupled into the layers did not show green upconversion luminescence to the bare eye, indicating that clustering effects of erbium ions are rather small.

SUMMARY

The etching behaviour of as-deposited reactively co-sputtered Al_2O_3 waveguide layers with low optical losses has been investigated using an inductively coupled reactive ion etch system and various process gases. Based on a comparison of the etch rates and patterning feasibility of prospective mask materials, a BCl_3/HBr plasma and and standard resist mask were selected to fabricate channel waveguides, resulting in optimized structures with well-defined, straight sidewalls. Etch depths of > 500 nm have been demonstrated, sufficient for channel waveguides with strong optical confinement and low bending losses. Propagation losses of around 1 dB/cm were measured in single-mode Al_2O_3 ridge waveguides defined using the developed etch process, indicating low added losses. Al_2O_3:Er layers fabricated using the same deposition method applied for the undoped layers show typical emission cross-sections, low green upconversion and

lifetimes of up to 7 ms. The combination of optimized deposition process and channel waveguide fabrication method will enable the patterning of highly compact active integrated devices in Al_2O_3:Er layers. Our future research will focus on channel waveguide light emitting structures employing this technology.

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