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High concentration optical doping of Al_2O_3 waveguide films by Er ion implantation

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Al_2O_3 films on oxidized Si substrates were optically doped by ion implantation with 800 keV Er ions to peak concentrations ranging from 0.01 to 3.6 at.%. The samples exhibit broad room-temperature photoluminescence around $\lambda = 1.533\ \mu m$, corresponding to intra-$4f$ transitions in Er^{3+}. The photoluminescence intensity increases roughly linearly with Er concentration, while the luminescence lifetime decreases from 8 ms at low Er concentrations to 4 ms at 1 at.%. At 3.6 at.% the lifetime is 6 ms. Cooperative upconversion effects are observed at high Er concentrations and pump powers.

I. INTRODUCTION

Erbium doping of planar optical waveguide materials has received considerable attention since the realization of Er-doped fiber amplifiers [1, 2]. An advantage of planar Er-doped amplifiers and lasers is that they may be easily integrated with other devices. For example, passive devices such as optical multiplexers and splitters are readily made in waveguide materials on Si substrates [3, 4]. The realization of planar optical amplifiers demands high concentrations ($\sim$ 1 at.%) of Er in the waveguide structures.

In this paper optical doping of Al_2O_3 with high concentrations of Er by ion implantation is studied. The use of ion implantation as a means for incorporating Er into waveguide materials has several advantages. Firstly, there is no limit to the Er concentration. Also, the implant profile has a Gaussian shape at a controllable depth, offering the possibility of matching the Er distribution to the optical mode profile in a waveguide. Lastly, ion implantation is a standard technique in silicon processing technology, and is compatible with the techniques used for defining waveguides. Several studies have concentrated on Er ion implantation into optical waveguide materials [5, 6, 7]; most of these dealing with silica-based materials.

Al_2O_3 is a promising candidate as host material for Er doping by means of ion implantation. The similarity in valency and lattice constants between Al_2O_3 and Er_2O_3 [8] may allow for incorporation of high concentrations of Er in the Al_2O_3 crystal structure. Also, Al_2O_3 is know to be a radiation hard material [9]. Furthermore, in view of its ionic nature, Al_2O_3 is an interesting material to study, as it may exhibit different properties than silica-based materials with respect to optical doping. Earlier work has shown that high quality, low-loss waveguide structures are readily made on silicon substrates using standard lithographic techniques [10, 11]. The relatively high-index Al_2O_3 waveguides cladded with SiO_2 confine light very well, making small device structures possible.

The photoluminescence (PL) intensity and lifetime of Er-implanted Al_2O_3 films is studied as function of Er concentration. The samples show clear, broad PL around 1.53 μm, measured at room-temperature. Concentration quenching and cooperative upconversion effects are observed in highly doped samples.

II. EXPERIMENTAL

The Al_2O_3 films were fabricated by radio-frequency magnetron sputtering of Al_2O_3 onto thermally oxidized Si (100) substrates (oxide thickness $\sim$ 2 μm). The process results in a cubic polycrystalline phase for the Al_2O_3 film. Details of the sputtering process and film properties can be found elsewhere [10, 11]. After deposition of a top SiO_2 cladding layer, single mode waveguides at 1.55 μm
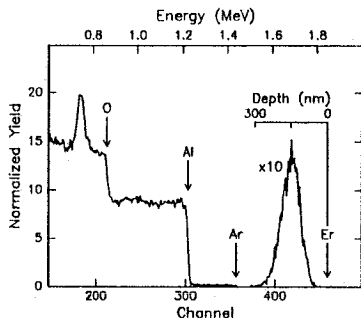
FIG. 1: RBS spectrum of an Al_2O_3 film implanted with 2.3×10^{15} 800 keV Er ions/cm^2.

FIG. 2: Room-temperature photoluminescence spectrum (spectral resolution = 2 nm) of an Er-implanted Al_2O_3 film (3.7×10^{15} Er/cm^2; peak concentration 3.6 at.%) annealed at 825 °C. A 514.5 nm excitation source at 50 mW was used. The inset shows the schematic energy level diagram for Er^{3+}.

can be realized. The optical loss of the waveguides is minimized to 0.35 dB/cm after thermal annealing at 825 °C for 1 hour in vacuum. Lateral confinement of the light may be achieved by etching a ridge in the guide prior to deposition of the top cladding [10, 11].

Al_2O_3 films without the top SiO_2 cladding layer were implanted with 800 keV Er to fluences in the range 1×10^{14} – 3.7×10^{16} ions/cm^2, with the samples held at room-temperature. Post-implantation thermal anneals at 825 and 950 °C were done in vacuum (base pressure $< 10^{-6}$ mbar) for 1 hour. The Al_2O_3 layer thickness and Er concentration profiles were measured by Rutherford backscattering spectrometry (RBS) using 2.0 MeV ^{4}He$^+$ and a scattering angle of 130°. Photoluminescence measurements were performed at room-temperature by exciting Er ions into the $^2H_{11/2}$ manifold using the 514.5 nm line of an Ar$^+$ ion laser. The pump light was directed onto the sample normal to the surface, and the pump power in the ~ 0.4 mm diameter spot was less than 500 mW in each case. The pump beam was mechanically chopped at 12 Hz. The luminescence was spectrally analyzed with a single-grating monochromator, again normal to the surface. The analyzed light was detected with a liquid-nitrogen-cooled germanium detector and the signal amplified using a lock-in amplifier. Lifetime measurements were performed by monitoring the decay of the luminescence after pumping to steady-state and mechanically switching off the light source (cutoff time ≈ 0.2 ms). The decay curves were averaged using a digitizing oscilloscope system.
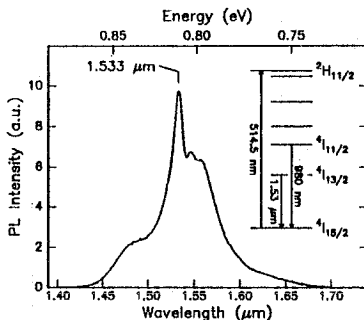
III. RESULTS AND DISCUSSION

Figure 1 shows the RBS spectrum of an Er-implanted Al_2O_3 film, implanted to an ion fluence of 2.3×10^{15} ions/cm^2. Analysis of the spectrum shows the Al_2O_3 layer thickness to be 430 nm. Furthermore, 0.4 at.% Ar is observed throughout the alumina film, due to the sputtering process. The Er profile is Gaussian, peaking at a depth of 140 nm with a full width at half maximum (FWHM) of 80 nm. The Er peak concentration determined from the spectrum is 0.23 at.%. Other samples implanted at different fluences showed Er peak concentrations in the range 0.01–3.6 at.%. Neither the Er profile nor the Ar concentration in the film changed significantly with annealing.

Figure 2 shows the PL spectrum of an Er-implanted sample (3.7×10^{16} ions/cm^2; peak concentration 3.6 at.%) after annealing at 825 °C. Our previous work has shown that thermal annealing at this temperature is necessary to optimize the PL intensity and lifetime [12], as well as the optical transparency of the waveguides [10, 11]. The spectrum peaks at 1.533 μm, corresponding to intra-4f transitions between the $^4I_{13/2}$ (first excited) state and the $^4I_{15/2}$ (ground) state of Er^{3+} [13]. The inset shows the schematic energy level diagram of the
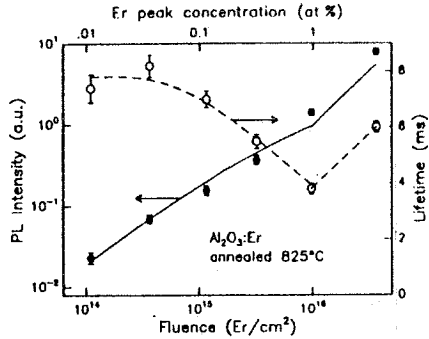
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FIG. 3: Photoluminescence peak intensity (left axis) and lifetime (right axis) as function of implanted Er fluence. The samples were annealed at 825 °C for 1 hour in vacuum. The corresponding Er peak concentration is plotted on the top axis. The dashed line through the lifetime data is a guide to the eye. The solid line is calculated from the dashed line using Eq. 1.

Er^{3+} free ion. The peak structure of the spectrum is attributed to Stark splitting of the degenerate $4f$ levels, characteristic of Er^{3+} embedded in a solid. This, as well as homogeneous and inhomogeneous broadening results in a FWHM of ~ 55 nm. Spectra for samples implanted at different fluences have different intensities but similar shapes, with small variations in relative intensity between the different lines.

The Er concentration dependence of PL intensity and lifetime is shown in Fig. 3. All samples were annealed at 825 °C. The bottom axis shows the Er implantation fluence on a logarithmic scale; the corresponding Er peak concentration is shown on the top axis. The PL intensity (filled data points, left axis) increases by a factor of 50 as the concentration is increased from 0.01 to 1 at.%. Above 1 at.% the intensity is seen to increase further. The lifetime (open data points, right axis) decreases slowly with increasing concentration from 8 ms at low concentrations to 4 ms at 1 at.%. At an Er peak concentration of 3.6 at.%, however, the lifetime increases again to 6 ms.

The behaviour of the PL intensity as function of Er concentration may be explained from the lifetime data. To a first order approximation (assuming that excitation into the $^2H_{11/2}$ manifold is followed

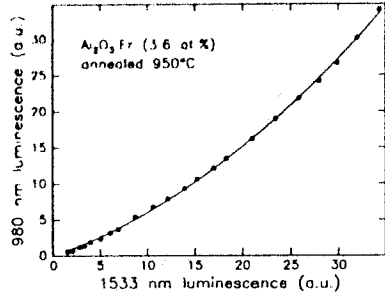FIG. 4: Photoluminescence intensity at 980 nm versus the luminescence at 1 533 μm for an Al_2O_3 film implanted with 3.7×10^{15} Er/cm^2 (peak concentration 3.6 at.%). The sample was annealed at 950 °C. The solid line is a second-order polynomial fit to the data

by rapid decay to the first excited state $^4I_{13/2}$ [13]) the PL intensity (I_{PL}) of the $^4I_{13/2} \rightarrow {}^4I_{15/2}$ transition can be written as

$$I_{PL} \propto f N \sigma_a I_{pump} \tau W_{rad} \qquad (1)$$

where f is the fraction of active Er ions, N the areal density (fluence) of Er ions, σ_a the pump absorption cross-section, I_{pump} the pump intensity, τ the measured lifetime, and W_{rad} the radiative decay rate. Non-radiative decay channels will lead to a reduction of the measured lifetime, decreasing the PL intensity. Assuming constant f, σ_a, and W_{rad}, it is possible to calculate the PL intensity (solid line) in Fig. 3 using the curve through the lifetime data (dashed line). The reasonable agreement with the intensity data suggests that the fraction of active Er ions (f) does not change with concentration up to ~ 1 at.%. The decrease in lifetime below 1 at.% is explained by an increase in the number of non-radiative decay channels, which may involve implantation-induced defects or concentration quenching [2] at high Er concentrations.

The unexpected behaviour at Er concentrations above 1 at.% is intriguing. One possible explanation that the high-fluence Er implant causes amorphization of the Al_2O_3. Indeed, earlier work on ion-implanted single crystal sapphire has shown that Al_2O_3 can be amorphized at fluences above $\sim 10^{16}$

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cm^{-2} [9]. Subsequent thermal annealing at temperatures around 800 °C causes recrystallization to a cubic phase of Al_2O_3. In this way the Er-doped Al_2O_3 films may also recrystallize, and thus might incorporate Er in a different fashion than in the case of low-fluence, non-amorphizing implants. This may lead to changes in lifetime and optically active fraction of the Er ions. Structral analysis of the (poly-crystalline) Er-implanted films is underway. The fact that relatively high lifetimes are obtained even at very high Er concentrations is very promising.

For the future application of Al_2O_3 films on a planar substrate as optical gain medium, the behaviour of lifetime and PL intensity under high pump intensities must be investigated. Preliminary results show that whereas the lifetime decreases only slightly with increasing pump power, the intensity depends sub-linearly on pump power. In addition, for high pump intensities, luminescence at 980 nm, corresponding to transitions from the $^4I_{11/2}$ manifold to the ground state (see inset Fig. 1), is observed. Figure 4 shows the the luminescence at 980 nm as function of the 1.533 μm luminescence for an Al_2O_3 film implanted to 3.7×10^{16} ions/cm^2 (Er peak concentration 3.6 at.%) after annealing at 950 °C. The solid line is a fit through the data using a second-order polynomial. This quadratic behaviour is indicative of cooperative upconversion [14], leading to less efficient pumping of Er to the $^4I_{13/2}$ level, and will be studied in more detail.

II. CONCLUSION

In conclusion, Er-implanted Al_2O_3 shows clear room-temperature photoluminescence at 1.53 μm. Lifetimes are as high as 8 ms at low Er concentrations. Increasing the Er concentration to 1 at.% lowers the lifetime to 4 ms. At 3.6 at.% the lifetime increases to 6 ms. The concentration dependence of the PL intensity is described by the lifetime as function of concentration. Upconversion processes can play a role at high Er concentrations and pump intensities. These highly doped Al_2O_3 films are promising candidates as optical gain media, which may be implemented in planar optical amplifiers and lasers in integrated optics.

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