Photoluminescence characterization of Er-implanted Al_2O_3 films

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Al_2O_3 films on oxidized Si substrates were implanted with 800 keV Er ions to peak concentrations ranging from 0.01 to 1 at. %. The samples show relatively broad photoluminescence spectra centered at $\lambda = 1.533$ μm, corresponding to intra-$4f$ transitions in Er^{3+}. At an Er peak concentration of 0.23 at. %, post-implantation thermal annealing up to 950 °C increases the photoluminescence intensity by a factor 40. This is a result of defect annealing, which increases the luminescence lifetime from 1 to 7 ms, as well as an increase in the Er^{3+} active fraction. High Er concentrations are achieved with only moderate concentration quenching effects.

Thin-film planar optical waveguides hold considerable potential in the field of integrated optics.[1,2] Recently, passive devices such as optical multiplexers and splitters have been fabricated in a variety of planar optical waveguide materials on Si substrates.[1,3,4] The next challenge is to dope such waveguide materials with optically active ions in order to realize planar active devices such as optical amplifiers and lasers. In particular, doping with the rare-earth Er is of interest because of its optical transitions at wavelengths around 1.54 μm, one of the standard wavelengths in optical telecommunications.

In this letter, optical doping of thin Al_2O_3 films by Er ion implantation is studied. Al_2O_3 is an interesting material for optical doping because waveguide fabrication technology is well developed for this material. Our earlier work has shown that high quality, low loss waveguide structures are readily made on silicon substrates using standard lithographic techniques.[5,6] The relatively high index Al_2O_3 waveguides cladded with SiO_2 confine light very well, making small device structures possible. Furthermore, the similarity in valency and lattice constants between Al_2O_3 and Er_2O_3[7] may allow for incorporation of high concentrations of Er in the Al_2O_3 crystal structure. Erbium implantation into different waveguide materials has been previously reported.[8,9] An advantage of ion implantation is the Gaussian implant profile at a controllable depth, offering the possibility of matching the Er distribution to the optical mode profile in a waveguide. Furthermore, ion implantation imposes no limit to the concentration, and is compatible with the other processing techniques for defining waveguides.

In this letter, the first successful incorporation of optically active Er in Al_2O_3 films is reported. Clear, broad photoluminescence (PL) at 1.53 μm is observed at room temperature, with luminescence lifetimes as high as 8 ms. The dependence of the PL intensity and lifetime is studied as a function of Er concentration and annealing temperature. Concentrations of up to 1 at. % may be reached without dramatic concentration quenching effects.

Al_2O_3 films were deposited on thermally oxidized Si(100) substrates by radio-frequency magnetron sputter-ing from an Al_2O_3 target. The SiO_2 thickness was ~ 6 μm and the Al_2O_3 thickness 600 nm. The deposition was carried out at a pressure of 0.8 Pa in an oxidizing ambient of 90% Ar and 10% O_2. The resulting structure of Al_2O_3 is a cubic polycrystalline phase. Details of the sputtering process and film properties can be found elsewhere.[5,6] Single-mode ridge waveguides have been realized using these films, and their optical loss can be minimized to 0.35 dB/cm by thermal annealing at 825 °C.

Erbium implantation was performed at 800 keV on uncladded Al_2O_3 films, held at room temperature. Implantation fluences ranged from 1×10^{14} to 1×10^{16} Er/cm^2. Post-implantation thermal anneals at 300–950 °C were done in vacuum (base pressure $< 10^{-6}$ mbar) for 1 h. The Al_2O_3 layer thickness and Er concentration profiles were measured by Rutherford backscattering spectrometry (RBS) using 2.0 MeV $^4He^+$ 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 diam 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.

The thickness of the Er-implanted Al_2O_3 films, as determined from RBS analysis (not shown), is 430 nm. Furthermore, 0.4 at. % Ar is observed throughout the alumina films, 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 concentrations were determined from the RBS spectra, and ranged from 0.01 to 1.0 at. % for all implanted samples.
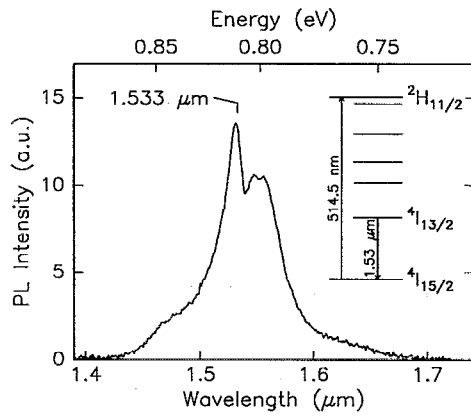
FIG. 1. Room-temperature photoluminescence spectrum (spectral resolution=2 nm) of an Er-implanted Al_2O_3 film (2.3×10^{15} Er/cm^2; peak concentration 0.23 at. %) annealed at 950 °C. A 514.5 nm excitation source at 100 mW was used. The inset shows the schematic energy level diagram for Er^{3+}.

Neither the Er profile nor the Ar concentration in the film changed significantly with annealing.

The PL spectrum of an Er-implanted sample (2.3×10^{15} ions/cm^2; peak concentration 0.23 at. %) after annealing at 950 °C is shown in Fig. 1. 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+}.[10] The inset shows the schematic energy level diagram of the 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 $\sim$55 nm. This is substantially broader than for Er-implanted SiO_2 (FWHM$\sim$11 nm)[8] or soda-lime silicate glass (FWHM$\sim$19 nm),[9] and offers larger bandwidth for multiplexed signal amplification. Spectra for samples implanted at different fluences and annealed at different temperatures have different intensities but similar shapes, with small variations in relative intensity between the different lines.

Figure 2(a) shows the PL peak intensity as a function of anneal temperature for samples with an Er peak concentration of 0.23 at. %. A gradual increase of the PL intensity is seen upon annealing up to 600 °C, after which the intensity is 6 times higher than for the as-implanted case. A further, and more rapid increase with temperature is seen above 600 °C, and another factor of 6 is gained after 950 °C annealing. The discontinuous behavior around 800 °C is reproducibly seen in different sample sets. The luminescence decay was also monitored, and found to be single-exponential in each case. Figure 2(b) shows the 1/e lifetime determined from the luminescence decay as a function of anneal temperature. The lifetime increases from $\sim$1 ms for the as-implanted case to 6–7 ms after annealing above 700 °C.

Photoluminescence intensity and lifetime are related through rate equations governing the excitation and decay of the Er ions. Assuming that excitation into the $^2H_{11/2}$ manifold is followed by rapid decay to the first excited
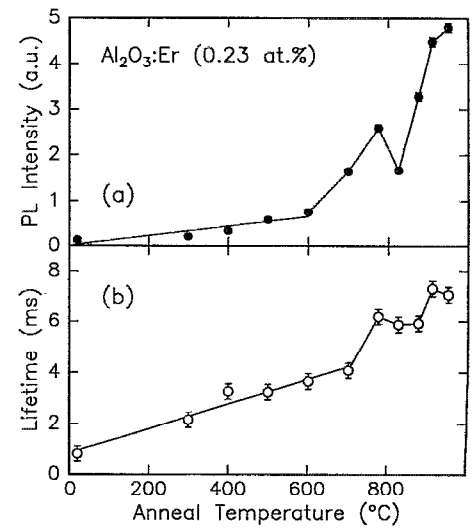

FIG. 2. Photoluminescence peak intensity (a) and lifetime (b) as a function of annealing temperature for an Al_2O_3 film implanted with 2.3×10^{15} Er/cm^2 (peak concentration 0.23 at. %). Thermal annealing was performed in vacuum for 1h. The solid lines are guides to the eye.

state ($^4I_{13/2}$), the PL intensity (I_{PL}) of the $^4I_{13/2} \rightarrow {}^4I_{15/2}$ transition can be written as

$$I_{PL} \propto fN\sigma_a(I_{pump}/h\nu)\frac{\tau}{\tau_{rad}} \qquad (1)$$

where f is the fraction of active Er ions, N the areal density (fluence) of Er ions, τ the measured lifetime, σ_a the pump absorption cross section, I_{pump} the pump intensity, and $h\nu$ the pump photon energy. The fraction τ/τ_{rad} is the ratio of the radiative decay rate to the total decay rate. Any non-radiative decay channels lead to a measured lifetime τ less than the radiative lifetime τ_{rad}.

The observed annealing behavior may be split into two different regimes. Below $\sim$700 °C, similar increases in I_{PL} and τ are seen, indicating that the increase in I_{PL} is predominantly caused by the increase in τ. This increase in τ is explained by a decrease in the number of nonradiative decay channels as a result of annealing of implantation-induced defects in the Al_2O_3 crystal structure. Indeed, previous studies have shown that temperatures up to $\sim$700 °C are required to anneal implantation-induced damage in Al_2O_3.[11,12] In the temperature regime above 700 °C, the increase in PL intensity is not accompanied by a strong increase in lifetime. According to Eq. (1), this suggests that f or σ_a increases, or that τ_{rad} decreases upon annealing. The latter is unlikely because this would cause a decrease of τ. Thus, assuming σ_a is constant, we explain the increase in intensity in terms of an increase in the fraction of optically active Er ions (f). The discontinuous behavior around 825 °C coincides with the attainment of minimum optical loss in Al_2O_3 waveguides, after annealing at 825 °C.[5,6] Apparently, the microstructural changes taking place around 825 °C affect the PL intensity.

Figure 3 shows the Er concentration dependence of PL intensity and lifetime after annealing at 825 °C. The bottom axis shows the Er implantation fluence on a logarithmic
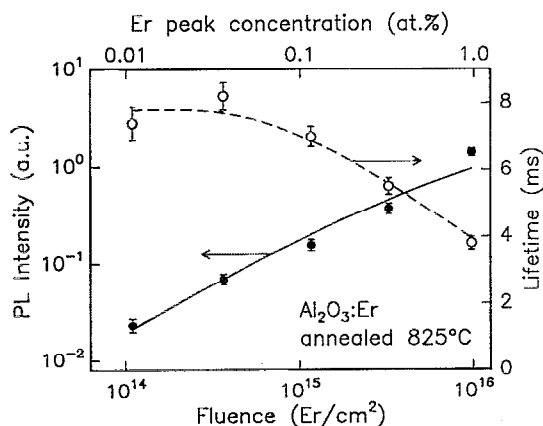
FIG. 3. Photoluminescence peak intensity (left-hand axis) and lifetime (right-hand axis) as function of implanted Er fluence. The samples were annealed at 825 °C for 1 h in vacuum. The corresponding Er peak concentration is plotted on the top axis. The dashed line through the lifetime data is a guide to the eye. The solid line is calculated from the dashed line using Eq. (1).

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. %. The lifetime (open data points, right axis) decreases slowly with increasing concentration from 8 to 4 ms. The sublinear increase in intensity is approximately accounted for by the decrease in lifetime. This becomes clear from a calculation of the PL intensity (solid line) using the curve through lifetime data (dashed line), employing Eq. (1) and using fixed f, τ_{rad}, and σ_a. The reasonable agreement with the intensity data suggests that the fraction of active Er ions (f) does not change with concentration. The decrease in lifetime is explained by an increase in the number of nonradiative decay channels, which may involved implantation-induced defects or concentration quenching[13] at high Er concentrations. The fact that relatively high lifetimes are obtained even at high Er concentrations is very promising. Note that for instance our earlier work on Er-implanted soda-lime-silicate glass showed a decrease in lifetime from 15 to 1.5 ms on increasing the concentration from .01 to 1 at%.[9] The difference may be explained by realizing that energy exchange between Er ions, which can lead to luminescence quenching, is less efficient in a material with widely spread Stark levels. Indeed, the PL spectra for Er-implanted Al_2O_3 are 2–3 times broader than those for the silicate glass.[9]

In conclusion, Er-implanted Al_2O_3 shows clear room-temperature luminescence at 1.53 μm. Thermal annealing at 950 °C increases the photoluminescence intensity by a factor of 40. The anneal behavior shows a low temperature regime below 700 °C where defect annealing takes place, and similar increases in intensity and lifetime are observed. Above this temperature the active fraction of Er ions increases, increasing the PL intensity further. Lifetimes are as high as 8 ms at low Er concentrations. Increasing the Er concentration to 1 at. % lowers the lifetime to 4 ms. These highly doped Al_2O_3 films are promising candidates as optical gain media, which may be implemented as planar optical amplifiers and lasers in integrated optics.

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