1.54 μm room-temperature luminescence of MeV erbium-implanted silica glass

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MeV erbium implantation doping of 10-μm-thick silica glass films on a Si substrate is studied with the aim of incorporating the rare-earth dopant on an optically active site in the silica network. As-implanted samples (3.5 MeV, 5×10^{15} Er ions/cm^2) show a strong luminescent transition at a wavelength of 1.54 μm, even at room temperature, corresponding to an intra-4f transition of Er^{3+}. Thermal annealing at temperatures up to 900 °C increases the luminescence intensity by a factor of 2 to 3. For temperatures above 1000 °C the intensity decreases drastically as a result of Er precipitation. The lifetime of the excited state is in the order of 10 ms. Photoluminescence studies at 4.2 K are used to identify implantation-induced damage.

Optical properties of rare-earth ions incorporated in glass hosts are of great interest in opto-electronic technology.[1] Erbium is of particular interest because of its intra-4f transition with a wavelength (λ) of 1.54 μm, coinciding with the low-loss window of standard optical telecommunications silica fiber. Recently, remarkable progress has been achieved in the development of single-mode Er-doped optical fiber amplifiers and lasers.[2–4] In this letter we present the first experiments on optical doping of planar μm-thick silica glass films deposited on Si substrates, using MeV erbium ion implantation. High-energy implantation has the important advantage that the implant profile can be accurately tuned on a μm depth scale, the typical dimension of optical waveguides and other integrated electro-optical structures. Indeed, MeV ion beams have previously been used to modify the refractive index of insulators and opto-electronic crystals with the aim of producing planar optical waveguides.[5] Experiments have been reported in the literature on Er implantation using medium-energy ion beams. These data concern implantation into Si,[6–8] III-V compound semiconductors,[8,9] and $LiNbO_3$.[10] We present the first room-temperature photoluminescence spectra of 3.5 MeV Er-implanted silica glass, showing a sharp and intense luminescent transition at a wavelength of 1.54 μm. The lifetime of the excited state is in the order of 10 ms. The annealing characteristics and diffusion and precipitation behavior are also studied.

10-μm-thick silica glass (amorphous SiO_2) films were deposited by thermal oxidation in a high-pressure steam ambient. This deposition process produces homogeneous low-loss silica films with a refractive index compatible with that of standard silica-based optical fiber.[11] Erbium implantation was performed using a 3.5 MeV Er^{++} beam from a 1.7 MV National Electrostatics Corporation tandem accelerator, with the samples at room temperature. The implantation fluence was 5×10^{15} ions/cm^2. The implantation profile was measured as a function of depth using Rutherford backscattering spectrometry (RBS) employing a 4.0 MeV He^{++} beam and a scattering angle of 170°, resulting in a depth resolution of 60 nm. Thermal annealing was carried out with a standard tube-furnace at a base pressure below 10^{-6} Torr. Cross-sectional transmission electron microscopy (TEM) was employed to study precipitation phenomena in some samples. Photoluminescence (PL) measurements were performed at room temperature and at 4.2 K, the latter by immersing the samples in liquid He. The 488 nm line of an Ar^+ laser was used as a pump source and luminescence spectra were detected using a single grating monochromator and a liquid-nitrogen-cooled germanium detector. All measurements were performed using the same pump power. The pump signal was mechanically chopped at 16 Hz and the signal was collected using a lock-in amplifier. Fluorescence decay measurements were performed using a 750 μs pump pulse also obtained by mechanical chopping.

Figure 1 shows a RBS spectrum for the as-implanted sample. The Gaussian-shaped Er profile peaks at a depth of 1.25 μm and has a full width at half maximum of 0.56 μm. The peak Er concentration is 0.10 at.%. The figure also shows a spectrum for an implanted sample annealed at 1200 °C for 1 h. As can be seen, no noticeable diffusion of Er is detected.

Figure 2(a) shows PL spectra measured at 4.2 K of an as-implanted sample, and of a sample annealed at 1000 °C for 1 h. The as-implanted sample shows a broad luminescence band over the full range of the spectrum. Superimposed on this band a peak is observed at $\lambda = 1.535$ μm (806 meV). The peak position of this line coincides with the wavelength of the transitions between the first excited manifold $^4I_{13/2}$ and the $^4I_{15/2}$ ground manifold of Er^{3+} ($4f^{11}$). After annealing at 1000 °C the broad band disappears and a narrow line is observed at $\lambda = 1.537$ μm. This line is highly asymmetric: a tail extends towards the high wavelength region. Earlier it has been shown that Er, when incorporated as a trivalent ion in a crystalline host, exhibits a fine structure of peaks in the PL spectrum on the high wavelength side of the main peak, due to different transitions between crystal field split levels.[6,8] As a result of

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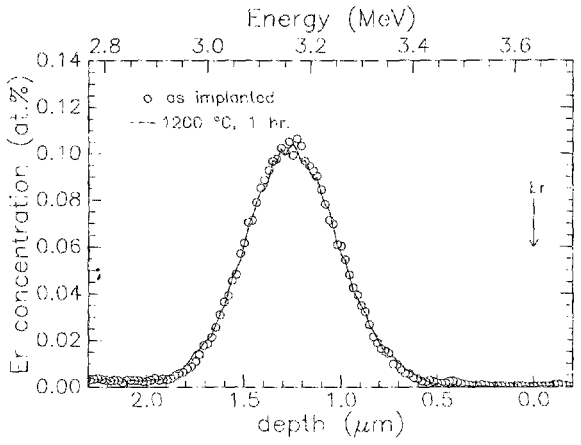

FIG. 1. RBS spectra of silica glass films implanted with 5×10^{15} 3.5 MeV Er ions/cm^2. Spectra for as-implanted (circles) and annealed (1200 °C, 1 h, drawn line) films are compared. The Er surface backscattering energy is indicated by an arrow.

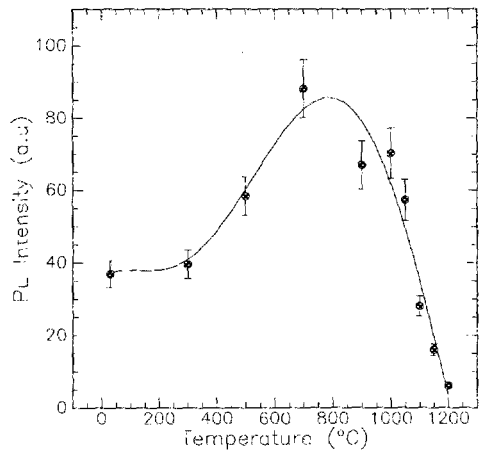

FIG. 3. Room-temperature photoluminescence intensity measured at $\lambda = 1.532\ \mu$m for different Er-implanted samples as a function of annealing temperature. The drawn line is a guide for the eye.

inhomogeneous and homogeneous broadening, this fine structure is not observed in the present amorphous glass host material. The broad luminescence band observed for the as-implanted sample is attributed to luminescence of implantation-induced defects in the silica network. The data show that annealing at 1000 °C suffices to anneal out these defects as far as their optical activity at 4.2 K is concerned.

Figure 2(b) shows PL spectra for the same two samples, recorded at room temperature. The broad defect band as observed for the as-implanted sample in Fig. 2(a) is not observed in this case. This can be explained by the fact that radiative transitions can be quenched at elevated temperatures as a result of an increased contribution of nonradiative transitions in the Er:silica system or through energy transfer from the excited Er^{3+} ions to other ions.[12] For the as-implanted sample a sharp PL peak is observed at $\lambda = 1.534\ \mu$m, with a lower intensity side peak at $\lambda = 1.550\ \mu$m. It is important to note that the as-implanted sample shows a measurable PL intensity at room temperature. This should be contrasted with results obtained for Er-implanted semiconductors in which case an annealing treatment is always necessary to obtain Er at an optically active site,[6,9] and in which case luminescence is often only observed at temperatures much lower than room temperature. This is probably a manifestation of the fact that the ionic bonding nature of the SiO_2 network provides the environment for erbium to be incorporated in its trivalent state, even when no annealing treatment is applied.

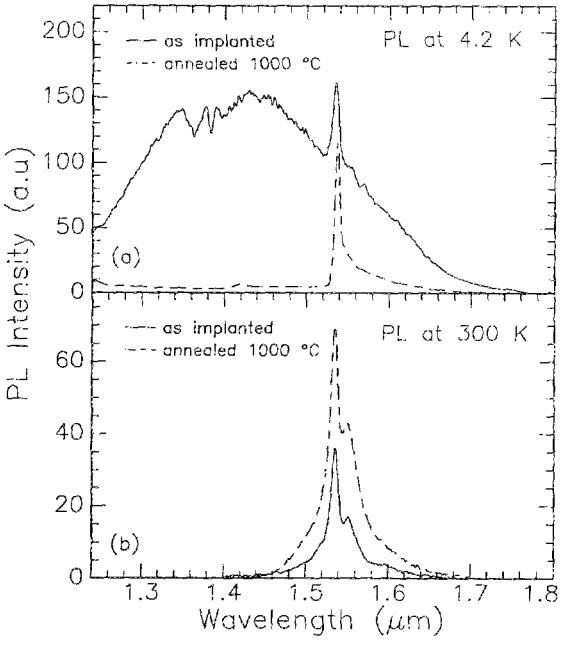

FIG. 2. Photoluminescence (PL) spectra for as-implanted (drawn line) and annealed (1000 °C, 1 h, dashed line) silica films. All measurements were performed using the same pump power (pump wavelength = 488 nm). (a) PL at 4.2 K, (b) PL at 300 K.

As can be seen in Fig. 2(b), thermal annealing at 1000 °C increases the PL intensity by a factor 2. Annealing does not change the overall shape of the spectrum. Figure 3 shows the room-temperature PL intensity as a function of annealing temperature. Intensities are measured at $\lambda = 1.532\ \mu$m for Er-implanted silica films annealed for 1 h at temperatures in the range 300–1200 °C. It should be noted that the PL intensity is determined by the overlap of the Er concentration profile and a standing wave pattern of the probe light in the glass layer. No absolute quantum efficiencies have yet been determined. The PL intensity increases with annealing temperature in the temperature range 300–700 °C. Earlier studies have shown that annealing temperatures above 400 °C are required to anneal implantation damage, such as dangling bond and vacancy complexes (e.g., E' and B_2 centers) in the silica network.[13–15] We therefore suggest that such damage is closely related to the Er optical activity. For temperatures above 1000 °C, a sharp decrease in PL intensity is observed. A preliminary investigation[16] using cross-sectional TEM of samples annealed at 1100–1200 °C shows a large concentration of Er-containing precipitates with a diameter of 10–20 nm. We therefore attribute the decrease in PL in-

tensity for temperatures above 1000 °C to these precipitation phenomena.

The lifetime of the $^4I_{13/2}$ excited state is a critical parameter for application of Er-implanted silica glass in waveguide lasers and amplifiers. Preliminary measurements have been performed of the room-temperature fluorescence decay at $\lambda = 1.534$ μm. The $1/e$ decay time for the as-implanted sample was found to be 8 ± 1 ms. This number is surprisingly high taking into account that no thermal treatment is applied to anneal out the implantation-induced defects. This shows once again that the glass network itself can accommodate these defects to a great extent. Thermal annealing was found to result in a further increase of the luminescence lifetime. A detailed lifetime study will be reported in a later publication.[17] These lifetimes are an order of magnitude longer than those observed in Er-implanted Si and III-V compound semiconductors[18] and, to our knowledge, are among the highest values ever reported for Er-doped silica glass.

This study shows that MeV ion implantation is a suitable technique to incorporate erbium on an optically active site in a silica network. PL measurements of as-implanted and thermally annealed samples show an intense luminescent transition at a wavelength of 1.54 μm, even at room temperature. Low-temperature PL data indicate the presence of irradiation-induced defects in the SiO_2 network. Thermal annealing at temperatures up to 900 °C can improve the room-temperature luminescence intensity by a factor of 2–3. For temperatures above 1000 °C precipitation effects play a role, reducing the PL intensity. The fluorescence lifetime is in the order of 10 ms, depending on implantation and annealing conditions. This long lifetime suggests that application in optical amplifiers and other integrated electro-optic devices might be possible.

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