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Tuning the Er³⁺ sensitization by Si nanoparticles in nanostructured as-grown Al₂O₃ films

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Nanostructured films consisting of single Si nanoparticles (NPs) and Er³⁺ ions layers separated by nanometer-scale Al₂O₃ layers of controlled thickness have been prepared in order to tune the energy transfer between Si NPs and Er³⁺ ions. The amorphous Si NPs with an effective diameter of ~ 4.5 nm are formed during growth and are able to sensitize the Er³⁺ ions efficiently with no postannealing treatments. The characteristic distance for energy transfer from Si NPs to Er³⁺ ions in Al₂O₃ is found to be in the 1 nm range. It is shown that in the nanostructured films, it is possible to achieve an optimized configuration in which almost all the Er³⁺ ions have the potential to be excited by the Si NPs. This result stresses the importance of controlling the dopant distribution at the nanoscale to achieve improved device performance. © 2009 American Institute of Physics. [DOI: 10.1063/1.3065520]

I. INTRODUCTION

Er-doped thin films are of great interest for the development of Si-compatible microphotonic devices such as integrated waveguide amplifiers for optical communications at 1.5 μm . The study of codoped Er³⁺-Si nanoparticle (NP) silica-based systems has attracted much attention because of the potential to combine the high absorption over a broad wavelength range of Si NPs with the efficient stable luminescence of Er³⁺. It has been demonstrated that in silica-based systems the Si NPs (usually nanocrystals) act as efficient sensitizers for Er³⁺. The interaction between the Si NPs and the Er³⁺ ions occurs on the nanometer scale and therefore is sensitive to the dopant distribution,¹⁻⁴ and can be affected by the presence of atomic defects.⁵ Usually, in silica-based systems, Si has to diffuse upon thermal treatment in order that the NPs precipitate from the initial mixed amorphous Si-SiO₂ structure and thus there is little control over the Si NP distribution.^{1-3,5} It is therefore desirable to develop preparation techniques that allow the formation of the Si NPs *in situ* during film growth. In addition, further understanding of the energy transfer from the Si NPs to the Er³⁺ ions may be achieved by studying the efficiency of such energy transfer in nonsilica-based materials. Of these, amorphous aluminum oxide (Al₂O₃) has been demonstrated to be an excellent host for Er³⁺ ions due to the high Er solubility and lower phonon energy compared to silica. Indeed a net optical gain has been reported for an Er³⁺-implanted Al₂O₃ waveguide prepared by sputter deposition.⁶ The formation of crystalline Si NPs in sapphire and in amorphous Al₂O₃ by different methods has been reported and has mainly focused on the study of the light emission from the Si NPs. However either weak or no Si NP-related light emission has been re-

ported, and its origin is still controversial.⁷⁻¹⁰ As a possible origin of the inefficient light emission of Si NPs in Al₂O₃ compared to silica based materials, it has been pointed out that the interface between the Si NPs and the Al₂O₃ tends to be abrupt and devoid of recombination centers, which have a high impact on the light emission properties.^{7,8} This inability of Si NPs to show efficient light emission when embedded in Al₂O₃ systems has so far limited the studies of Er³⁺ sensitization by Si NPs.

In this work we report efficient Er³⁺ sensitization by Si NPs embedded in nanostructured amorphous Al₂O₃ films. The films have been structured in the growth direction in order to maintain the Er³⁺ ions and the Si NPs in well-defined layers with a controlled separation at the nanoscale, and thus to tune the energy transfer from the Si NPs to the Er³⁺. The Si NPs are formed *in situ* during deposition, and the films have not been annealed after deposition. As a result the NPs remain amorphous and the film is not modified by diffusion processes that might modify the original structure, as in most previous reports.^{1-3,5} This procedure opens a route to the development of one step low-temperature processing for codoped nanostructured waveguide devices.

II. EXPERIMENT

The films have been prepared by alternating pulsed laser deposition (PLD) in vacuum ($P \sim 4 \times 10^{-6}$ Torr) onto chemically cleaned Si (100) and fused silica substrates held at room temperature. An ArF excimer laser ($\lambda = 193$ nm, $t = 20$ ns full width at half maximum) was focused alternately on independent metallic Er, Si, and ceramic Al₂O₃ targets with an energy density of 2 J cm⁻². In this way the deposition conditions for each target are controlled separately.^{11,12} The schematic structure of the films is shown in the inset of Fig. 1. Each film consists of nine Si NP single layers, each of which is sandwiched between five Er³⁺ layers on each side.

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FIG. 1. RBS spectrum corresponding to the film with $s=0$ nm. The surface channels of the different elements in the film are indicated. The inset shows a schematic of the film distribution of Er^{3+} ions and Si NPs in the growth direction.

The Er^{3+} layers are separated between them by 7 nm thick Al_2O_3 layers in order to minimize quenching of the luminescence due to Er^{3+} – Er^{3+} interaction.¹² Films with different nominal separations ($s=0, 4, 7, 11$ nm) between the Si NP layer and the first Er^{3+} layer on either side have been prepared by depositing a controlled amount of Al_2O_3 . Note that the Er^{3+} ions are contained in the same layer as the Si NPs for the $s=0$ nm film. A single pulse on the Er target has been used to deposit the Er^{3+} ions.^{11,12} For the Si deposition 500 pulses on the Si target have been used in order to induce the formation of Si NPs *in situ* following a Wolmer–Weber growth mode, as has been reported for metal NPs such as Cu and Ag.^{13,14} Finally, in order to obtain films with the same total thickness, and thus to compensate for the different values of s in each film, two Al_2O_3 buffer layers of thickness t have been deposited, one next to the substrate and another capping the film. The schematic structure of the film starting from the substrate can be described as substrate/ t - Al_2O_3 / $9x[5 \times (\text{Al}_2\text{O}_3:\text{Er}) / s\text{-}\text{Al}_2\text{O}_3/\text{Si-NPs}/s\text{-}\text{Al}_2\text{O}_3]$ / $5 \times (\text{Al}_2\text{O}_3:\text{Er}) / t\text{-}\text{Al}_2\text{O}_3$. For comparison purposes two reference films have been prepared: a film doped only with Er and a film doped only with Si NPs, both with the same spatial distribution as the film with $s=11$ nm.

The Er- and Si-concentration depth profiles were determined by Rutherford backscattering spectrometry (RBS) using a 1.97 MeV $^4\text{He}^+$ beam and a dispersion angle of 165° . The structural analysis of the sample was performed by transmission electron microscopy (TEM) analysis. Cross-section TEM samples were prepared using a “lift-out” focused ion beam specimen preparation technique. The samples were observed in a Tecnai F20ST TEM operated at 200 kV with a point-to-point resolution of 0.24 nm. Energy-filtered TEM (EFTEM) images of the samples were obtained by filtering in the low-loss region of the electron energy loss spectrum at the plasmon peak corresponding to pure Si, centered at about 16 eV, using a window width of 4 eV. In this way EFTEM can be used to obtain composition maps of the Si-rich areas in which the Si NPs can be observed, independent of their crystalline or amorphous structure.¹⁵

Optical transmission measurements have been per-

FIG. 2. EFTEM cross-section image corresponding to the film with $s=11$ nm. The image has been recorded filtering at the plasmon peak corresponding to pure Si at energy of 16 eV. The light color areas correspond to the Si NPs layers.

formed in the 300–1700 nm wavelength range. From these measurements the absorption spectra of the films have been obtained in optical density units [$\text{OD}=\log_{10}(1/T)$]. Photoluminescence (PL) measurements were performed at room temperature using a single grating monochromator and standard lock-in techniques with a Hamamatsu photomultiplier in the near infrared (900–1700 nm). The excitation was performed at 476.5 and 514.5 nm using an Ar^+ ion laser. Further details can be found elsewhere.^{11,12}

III. RESULTS AND DISCUSSION

A. Structural analysis

The RBS spectrum from the film with $s=11$ nm is shown in Fig. 1. In the low energy region of the spectrum, the (Al+Si) signal contribution shows oscillations that are associated with the layered structure of the film. In the Al signal region of the spectrum, nine minima are observed, which correspond to the presence of the Si NP layers. Detailed quantitative interpretation of the RBS spectra was undertaken with assistance of the SIMRA code. The analysis shows that the Er areal density per layer is $3.4 \times 10^{13} \text{ cm}^{-2}$, which is similar to that reported in our previous work on $\text{Al}_2\text{O}_3:\text{Er}$ films.^{11,12} The Si areal density per layer is $7.0 \times 10^{15} \text{ cm}^{-2}$, which is equivalent to an effective thickness of 1.4 nm, taking into account the density of the $\alpha\text{-Si}$.¹⁶ The total film thickness is 510 ± 10 nm and the s values determined for the films are 0, 4.0, 7.0, and 11.0 ± 0.5 nm, showing that a fine control of the deposited Al_2O_3 has been achieved.

Figure 2 shows an EFTEM image corresponding to the cross section of the film with $s=11$ nm, filtered at the Si plasmon peak: The light contrast lines in the image correspond to the Si-rich regions in the film. The contrast in these regions is not uniform, indicating that they are not continuous but rather are formed by Si NPs. The darker background corresponds to the Al_2O_3 layers. Note that the background is not completely dark due to the fact that the Al plasmon peak in Al_2O_3 is close to that of the Si plasmon peak in pure Si. The low doping concentration of Er prevents its observation. The layers are well defined and uniform, and the spacing

FIG. 3. Absorption spectra of the Al_2O_3 film doped only with Er and the Al_2O_3 film doped only with Si NPs as a function of the photon energy (bottom) and wavelength (top).

between the layers is in excellent agreement with that determined from the RBS analysis. High resolution TEM analysis shows that there is no crystalline structure in either the Al_2O_3 layers or the Si-rich areas, indicating the amorphous nature of both the Al_2O_3 and the Si NPs. Analysis of the EFTEM images allows the thickness of the Si-rich layers to be determined as 4.5 ± 0.5 nm. It can be assumed that this value is related to the diameter of the Si NPs in the growth direction. This diameter is consistent with our previous reports for metal NPs prepared by PLD for a similar areal density of deposited material, taking into account the differences in density.^{13,14}

Figure 3 shows the absorption spectra measured for the reference films deposited on fused silica, and doped only with either Er or Si NPs. The oscillations are due to the light interference between the film surface and the film-substrate interface. The reference film doped only with Er shows a negligible absorption in the whole photon energy range. The spectrum for the reference film doped only with Si NPs shows also a negligible absorption in the infrared region and a clear absorption band in the visible region above 2 eV, due to the presence of Si NPs. The absorption spectra for all the films codoped with Er and Si NPs are similar to that of the reference film doped only with Si NPs, further confirming that all films have the same Si content and linear optical properties. The bandgap energy associated with the Si NPs has been estimated by assuming a dependence for an indirect bandgap semiconductor and fitting the absorption spectrum to the expression $(\alpha h\nu)^{1/2} \sim (h\nu - E_{\text{og}})$, where α , $h\nu$, and E_{og} are the absorption coefficient, the incident radiation energy, and the optical bandgap energy, respectively. The value obtained for E_{og} is 1.7 eV. Reports on experimental measurements and calculations of E_{og} as a function of the size of Si NPs embedded in SiO_2 show that the value obtained corresponds to NPs with an average diameter in the range of 4–5 nm.^{17–20} This result is in very good agreement with the results obtained from the EFTEM analysis, and therefore for analysis purposes from now on it will be considered that the average diameter of the Si NPs in the films is 4.5 ± 0.5 nm.

FIG. 4. (Color online) PL spectra for Er^{3+} and Si NP codoped Al_2O_3 films with different values of s , and for the films doped only with Er or with Si NPs. The value of s is indicated on the corresponding spectrum. The pump power used was 500 mW under nonresonant (476.5 nm) excitation conditions.

B. PL enhancement

The PL spectra excited at 476.5 nm are shown in Fig. 4. The reference film doped only with Er shows no Er^{3+} related emission around 1540 nm, in agreement with the fact that at 476.5 nm there is no resonant Er^{3+} energy level for absorption. An Er^{3+} -related signal is also not seen for the films codoped with Si NPs and Er^{3+} , and with $s=7$ and 11 nm. In fact, the spectra for these two films are similar to those of the reference film doped only with Si NPs. The spectra show a flat weak band extending from 1400 to 1650 nm, which is related to the presence of Si NPs. As stated in Sec. I, the origin of light emission from Al_2O_3 containing Si NPs is very controversial,^{7–9} and its understanding needs further analysis that is outside the focus of this work. In contrast, the film with $s=0$ nm shows a clear strong Er^{3+} emission with the maximum at 1540 nm. The film with $s=4$ nm also shows a small Er^{3+} -related signal. This result is an evidence for energy transfer from the amorphous Si NPs to the Er ions for the films with $s=0$ and 4 nm. Under excitation at 514.5 nm (resonant excitation on the Er^{3+} ions) all the Er-doped films show the characteristic Er^{3+} emission.

In order to analyze the results, Fig. 5 shows the PL intensity of the films at 1540 nm as a function of s , for excitation at 476.4 and 514.5 nm. It can be observed that for both excitation wavelengths the PL intensity decreases sharply as s increases. For reference, the PL intensity for the reference film doped only with Er at an excitation wavelength of 514.5 nm is also shown on the y-axis. The PL intensity value for the film doped only with Er is very weak, in agreement with our previous reports for $\text{Er}:\text{Al}_2\text{O}_3$ films which show that in the as-grown condition the films exhibit a negligible emission, and that it is necessary to anneal the films at temperatures higher than 750 °C for 1 h to achieve a significant signal.^{11,12} For excitation at 514.5 nm the film with $s=0$ shows an emission intensity that is one order of magnitude larger than that of the film doped only with Er. In Fig. 4 it can be seen that emission under excitation at 514.5 nm is

FIG. 5. (Color online) PL intensity at 1540 nm as a function of s , excited at 476.5 nm (full circles) and 514.5 nm (open circles). The PL intensity for the film doped only with Er excited at 514.5 nm is represented on the y-axis with a full triangle.

slightly higher than that under excitation at 476.5 nm. This can be due to the additional emission of directly pumped Er^{3+} ions, i.e., those that cannot be excited through Si NPs. Indeed, due to the controlled multilayer doping of the films it can be shown that not all Er^{3+} layers can be effectively excited by the Si NPs. For the film with $s=7$ nm no Er^{3+} PL-related emission is observed under pumping at 476.5 nm, which suggests that for the film with $s=0$ nm only the Er^{3+} layers in close contact contribute to the sensitized emission, since the next nearest Er^{3+} layers in the $\text{Er}:\text{Al}_2\text{O}_3$ doped region have been deposited at a distance of 7 nm from the Si NP layer due to the film design. This means that only 18 layers out of the total of 50 Er^{3+} have the potential to be sensitized, i.e., only 36% of the total Er in the film contributes to the sensitized signal.

The results presented have interesting technological implications since they demonstrate that amorphous Si NPs formed *in situ* during the growth process are able to efficiently transfer energy to Er^{3+} ions. This shows that there is no need for thermal treatment to activate the energy transfer, and opens the possibility of fabricating devices codoped with Er–Si NPs at low temperature. Note that although it has recently been confirmed that amorphous Si NPs can sensitize Er^{3+} ions to enhance both the Er^{3+} PL and electroluminescence,²¹ annealing at temperatures in the range of 800–900 °C has been always required. Furthermore, in spite of the presence of defects in the as-grown Al_2O_3 films, there is evidence of significant energy transfer, indicating that although defects might modify the efficiency of the energy transfer process, as has been suggested in the case of Si-rich silica films,⁵ they do not preclude it.

C. Si NP-Er energy transfer: Critical distance and fraction of sensitized ions

The results show that the controlled nanostructure of the films allows the energy transfer from the Si NPs to the Er^{3+} ions to be effectively tuned. It is also possible to estimate the

critical distance for energy transfer from the surface of a Si NP to Er^{3+} in Al_2O_3 . For this purpose it is important to analyze in detail the structure of the film with $s=4$ nm. In this film there is still clear evidence of excitation through the Si NPs. The two Er^{3+} layers nearest to each Si NP layer have to be considered separately because the effective Si NP- Er^{3+} separation is different in each case. For the Er^{3+} layer deposited before the Si NPs it is a good approximation to assume that the Er^{3+} –Si NP separation is the thickness of the deposited Al_2O_3 layer, i.e., 4 nm. This separation will be the *minimum separation* between an Er^{3+} ion and the lower surface of the subsequently deposited Si NP. In order to estimate the Si NP- Er^{3+} separation for the Er^{3+} layer deposited after the Si NPs, the thickness of the deposited Al_2O_3 layer must be considered, and also the volume of the Si NPs that is embedded within the Al_2O_3 . It can be assumed that the total thickness of the Al_2O_3 with embedded Si NPs is the sum of the effective thickness of the Si NPs (1.4 nm) plus the effective deposited 4 nm thick Al_2O_3 layer, which yields a total thickness of ~ 5.4 nm. Since the NPs have an average diameter of 4.5 ± 0.5 nm, then the NPs are totally covered before the next Er^{3+} layer is deposited. In this case the minimum separation of an Er^{3+} ion to the upper surface of a Si NP underneath will be of ~ 1 nm. For Er^{3+} in silica-based systems characteristic energy transfer distances from 0.5 to 2–3 nm have been reported,^{1–3} and therefore for the film with $s=4$ nm it is suggested that there is negligible energy transfer from the Si NPs to the Er^{3+} layers deposited before the Si NPs, which are at a separation of 4 nm, and that the effective energy transfer is mainly due to the Er^{3+} layer deposited after each Si NP layer. From these results it can thus be estimated that there is effective energy transfer from Si NPs to Er^{3+} ions in Al_2O_3 for a distance of ~ 1 nm.

Regarding the fraction of Er^{3+} ions that can be excited via Si NPs in the nanostructured film, an estimation can be performed for the film with $s=0$ nm. It is important to realize that for this film the Er^{3+} layers deposited after and before the Si NPs are effectively in the same layer together with the Si NPs. Thus for this case the areal density of Er^{3+} ions around the Si NPs is twice the Er areal density per layer determined from the RBS measurements, namely, $3.4 \times 10^{13} \text{ cm}^{-2}$. The areal density of Si NPs per layer can be estimated as $[\text{Si}]/V_{\text{NP}} \times \rho$, where $[\text{Si}]$ is the areal density of Si as determined from the RBS measurements, V_{NP} is the volume of an average NP calculated by assuming that they are spheres with an average diameter of 4.5 ± 0.5 nm, and ρ is the density of amorphous Si.¹⁶ The result of the calculation yields an areal density of NPs of $\sim 3 \times 10^{12} \text{ cm}^{-2}$, which is one order of magnitude less than the areal density of Er^{3+} ions. Nevertheless the results should be analyzed taking into account the two-dimensional distribution in the Si NPs and the Er^{3+} ion layer. Assuming a diameter of 4.5 nm for the NPs it can be determined that 99.5% of the Er^{3+} ions are within an interaction range of 1 nm from the surface of a Si NP. Therefore almost all the Er^{3+} ions in the layer have the potential to be effectively excited by a Si NP. In fact in order to excite all the Er^{3+} in the layer each Si NP should be able to excite about 22 Er^{3+} ions, which is close to the optimum coupling conditions estimated for Er^{3+} in Si-rich SiO_2 .²

IV. CONCLUSIONS

Efficient energy transfer from Si NPs to Er^{3+} ions in a nonsilica-based material Al_2O_3 has been demonstrated. The Si NPs are amorphous and are formed *in situ* during deposition by PLD at room temperature; they are readily able to transfer energy efficiently to the Er^{3+} ions. Efficient energy transfer between Si NPs and Er^{3+} ions without the need for postdeposition annealing is demonstrated. This result opens a possible route for the fabrication of low-temperature low-cost Er-doped waveguide devices. The spatial distribution of the Er^{3+} and Si NPs has been controlled at the nanoscale, and it has been shown that the energy transfer depends critically on the Si NP- Er^{3+} separation, and is optimized for a characteristic interaction distance of ~ 1 nm. The control of the spatial distribution of the Er^{3+} ions around the Si NPs at the nanoscale that has been achieved allows the amount of Er^{3+} sensitized by Si NPs to be effectively tuned. The present results imply that a higher percentage of Er^{3+} ions can be sensitized by Si NPs if structures are prepared with one Er^{3+} layer per Si NPs layer, for which the Er^{3+} /Si NPs ratio is optimized.

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