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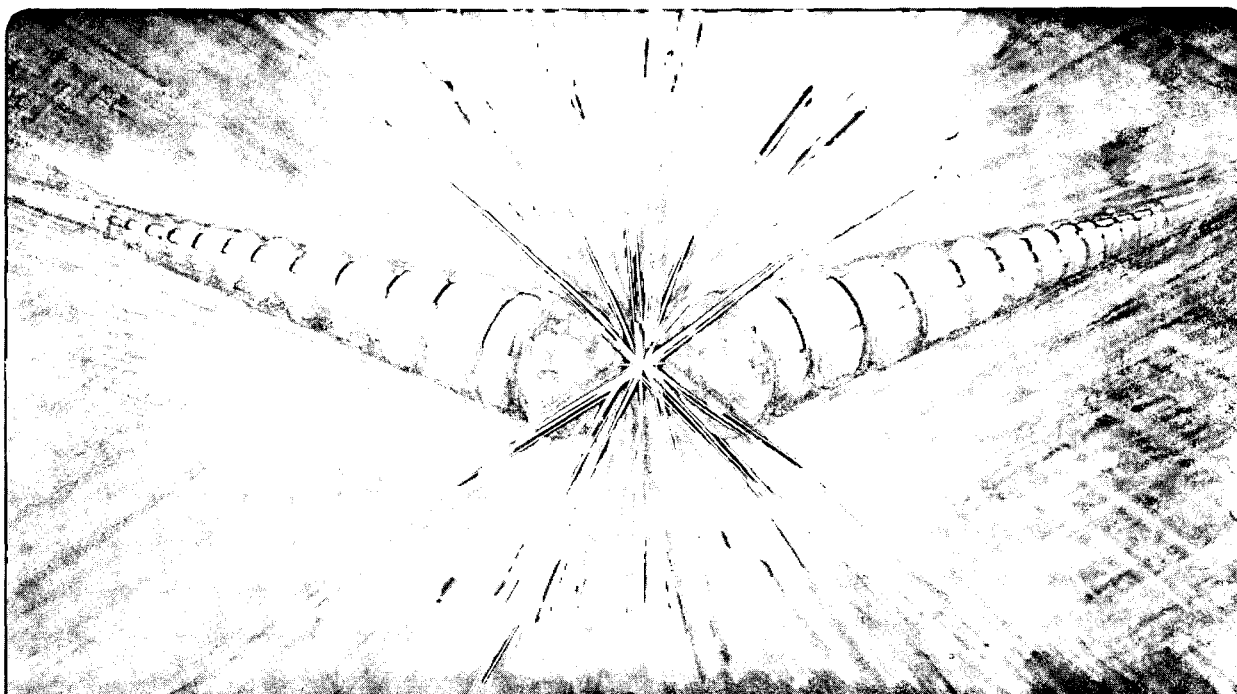
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March 1995



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High Concentration Erbium Implantation of Epitaxially Grown CaF₂/Si Structures*

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HIGH CONCENTRATION ERBIUM IMPLANTATION OF EPITAXIALLY GROWN CaF_2/Si STRUCTURES.

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ABSTRACT

Calcium fluoride thin films grown on silicon substrates by sublimation under ultra high vacuum are well known to be highly efficient hosts for rare earth luminescence properties. For this reason we incorporate erbium by ion implantation in order to form optoelectronic integrated devices. Here we describe the incorporation conditions of erbium in CaF_2/Si structures and their luminescence characteristics. The properties of the material have been investigated for implantation doses varying from 4×10^{14} to 1×10^{17} at.cm⁻². The role of oxygen in the charge compensation mechanisms is investigated and it is shown that the maximum emission in erbium at 1.53 μm occurs for an implanted dose of 2×10^{16} at.cm⁻². This corresponds to an Er concentration three orders of magnitude greater than for the case of classical-erbium-doped semiconductors. At this high concentration (up to 15 at.%) the light emission mechanisms are of great theoretical interest. They involve strong Er-Er coupling effects: energy transfer, cross-relaxation phenomena and high conversion efficiencies.

These properties make erbium-implanted CaF_2/Si structures excellent candidates for the production of optically active waveguides. The guiding structure can be formed by high energy implantation to build a buried active region of high refractive index within the CaF_2 thin film.

INTRODUCTION

Incorporation of trivalent rare-earth (RE) ions in waveguides holds considerable potential in the field of new processes for optical telecommunication systems. Among REs, erbium is of particular interest because of its $^4I_{13/2} \rightarrow ^4I_{15/2}$ intra-4f transition with a wavelength of 1.53 μm , coinciding with the low-loss window of standard optical telecommunications silica-based fibers¹. Remarkable progress has been achieved in the development of Er-doped optical fiber amplifiers and lasers². Our ultimate goal however, is the production of integrated high gain amplifiers or laser devices on the centimeter scale. Recently, erbium-doped waveguide materials integrated on semiconductor substrates have been investigated and have shown amplification and laser properties^{3,4}.

The realization of optoelectronic integrated circuits⁵ (OEICs) such as active planar optical amplifiers, multiplexers or splitters requires high concentration of luminescent centers in the guiding region; a parameter directly related to amplification performances. Dielectric materials are suitable for the incorporation of high RE concentration with long luminescence lifetimes. Furthermore, they can be grown as thin films on various semiconductor substrates to form true optical-integrated devices⁶⁻¹¹. In a previous study^{12,13} we have shown that epitaxial $\text{Ca}_{1-x}\text{Er}_x\text{F}_{2+x}/\text{Si}$ thin films exhibit high intensity 1.53 μm emission at room temperature. In this material, the erbium concentration (x) can be as high as x=16% without quenching of the 1.53 μm luminescence. These epitaxial structures were formed by sublimation of $\text{Ca}_{1-x}\text{Er}_x\text{F}_{2+x}$ powders under ultra high vacuum. Due to the particular thermodynamic conditions of the growth technique, the erbium ions were found to be in highly symmetric sites, involving sharp emission lines and long luminescence lifetimes.

In this paper, we report on the incorporation of erbium by ion implantation in CaF_2/Si epitaxial thin films. The basic idea is to increase the oscillator strength of the intra-4f¹¹ transitions (and consequently the luminescence intensity), by incorporating the Er^{3+} ions in "parasitic" sites, while keeping a high active center concentration.

Er IMPLANTATION AT HIGH DOSES: A NON-EQUILIBRIUM SYNTHESIS.

The CaF_2 thin films were epitaxially grown at LEMME, University of Bordeaux-France, on Si (100) substrates by sublimation under ultra-high-vacuum. The conventionally-cleaned substrates (degreasing, etching, deoxidization under a dry nitrogen atmosphere) were outgassed at 800°C at a pressure of 5×10^{-11} Torr. The surface reconstruction was followed by Rapid High Energy Electron Diffraction (RHEED). The initial CaF_2 powder was evaporated at 1250°C from platinum crucibles; the growth rate was $0.1 \text{ nm} \cdot \text{s}^{-1}$ and the temperature of the substrate 550°C . Under these conditions, thin films of good epitaxial quality can be obtained, and Rutherford Backscattering Spectroscopy (RBS) measurements in channeling conditions have revealed a χ_{min} of 4% for 150nm-thick thin films¹⁴.

The Er implantation was performed using a vacuum-arc-based implanter¹⁵ developed at LBL, University of California, with an average beam energy of 150 KeV and an ion beam current density of $5 \text{ mA} \cdot \text{cm}^{-2}$. The source neither requires nor produces an ambient gas for its operation, and is typically run in a vacuum of around $P=1 \times 10^{-6}$ Torr. The source was pulsed, with a pulse duration of 250 μs and a repetition rate of 1 to 4 pps. Under these conditions, an implantation dose of $10^{16} \text{ at} \cdot \text{cm}^{-2}$ can readily be achieved in a time of a few minutes.

As an ion enters the target, various interactions with the latter slow down the incident particle. The depth of implantation is determined by the ion energy and mass and the substrate atomic species. Two kinds of interactions come into play: (i) electronic stopping occurs for highly energetic incoming ions (MeV) and does not generally affect the crystallinity of the target. For the energy range presently involved (150 keV), the most important interaction is (ii) the nuclear one, assuming coulomb-interactions between the nuclei of the incident ion and the atoms of the target. This process severely modifies the local order in an implanted crystal. Incident ions are scattered by the target atoms which can be recoiled to depth. As a result, numerous defects are created such as crystalline dislocations, amorphization, stoichiometric and valence disturbances, etc. However, for ionic crystals (CaF_2 for example) the amorphization implantation-dose is very high¹⁶ ($\geq 10^{17} \text{ ions} \cdot \text{cm}^{-2}$). All these phenomena jointly determine the final implantation profile.

In the ideal and simplified low-dose case, the depth distribution can be approximated by a gaussian, and then the mean of the distribution is the "projected range" R_p , and the concentration of the implanted species. For high implantation doses two new factors come into play: (i) the increase in density of the material and changes of composition; for Er-implanted CaF_2 , the previously implanted Er ions result in a global increasing of the nuclear stopping power of the target, and consequently modifies the initial gaussian shape. For high doses and high incident beam currents, as here, (ii) the target can also be sputtered. We will see that this last effect is of great importance in the case of Er-implanted CaF_2/Si epitaxial structures.

In figure 1, we show the evolution of the projected ranges and the erbium atomic fraction at the maximum of the implanted profile as a function of the erbium incident implantation dose. These values were obtained by RBS measurements performed with a 1.8 MeV $^4\text{He}^+$ beam. The integrated charge was $10 \mu\text{Cb}$ and the scattering angle $\theta_1 = 105^\circ$ in the laboratory coordinates.

As can be seen on figure 1, increasing the erbium implantation dose leads to the reduction in R_p , which decreases from a maximum of 400Å to a minimum of 150Å. This is attributed to sputtering of the CaF_2 thin film by the ion beam that etches away the surface of the previously-implanted material. For the highest dose ($10^{17} \text{ at} \cdot \text{cm}^{-2}$), the thickness that has been eaten away can be estimated as 330Å. The full line curve

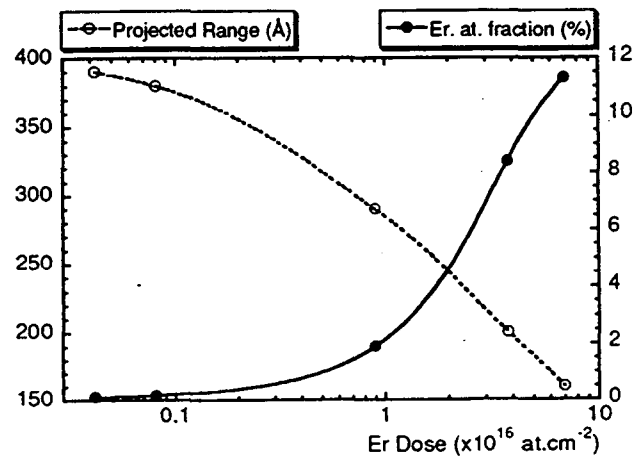


Figure 1 - Erbium implanted CaF_2/Si (150 keV) Evolution of the projected range (dashed line) and the erbium atomic fraction at the maximum of the implantation profile (full line).

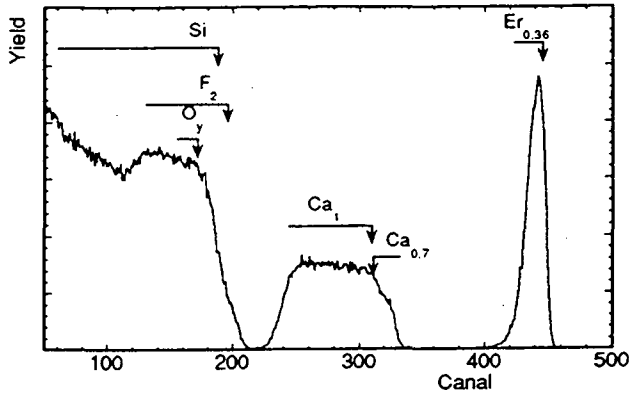
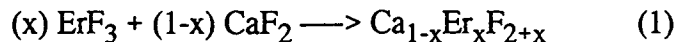


Figure 2 - RBS spectrum of an Er-implanted CaF₂/Si structure. Er dose: $6.3 \times 10^{16} \text{cm}^{-2}$ - $E=150 \text{keV}$

indicates the erbium atomic fraction at the maximum of the implanted profile. Because of the sputtering phenomenon, the retained dose is lower than the incident one. This should not be critical for the production of integrated waveguide structures; for this type of applications, the implanted region must be buried typically $1 \mu\text{m}$ below the surface, involving higher implantation energies (MeV). At such energies, the significant interaction phenomenon at the surface is electronic stopping, which does not cause sputtering of the target.

With these considerations, it is of interest to understand the local order in the implanted thin films and its implications on the luminescence properties. Let us keep in mind that during the implantation process, the system is in a non equilibrium state concerning the charge compensation process: we introduce a trivalent rare earth ion (Er^{3+}) at high dose in a divalent matrix ($\text{Ca}^{2+} + 2\text{F}^-$), and the conservation of electric charge must be assured by one at least of the following mechanism^{17,18}: (i) Charge compensation by interstitial F^- ions that have been displaced from their original position because of recoil processes; (ii) electronic charge compensation that could involve valence-state disturbances, and creation of "hole-type" defects, or (iii) charge compensation by interstitial O^{2-} ions diffusing in the film during annealing.

Figure 2 shows an RBS spectrum of a CaF₂:Er/Si thin film (implanted dose: $6.3 \times 10^{16} \text{at.cm}^{-2}$). The structure was annealed in air (20.95% O₂ content) at $T=500^\circ\text{C}$ for one hour. The thickness of the initial CaF₂ layer is 4300\AA , the depth of the maximum concentration is 160\AA and the straggling of the Er profile is 530\AA . The composition of the unimplanted region is found to be $\text{Ca}/\text{F}=1/2$. In the implanted region however, at the peak concentration, the composition of the material is estimated to be $\text{Ca}_{0.7 \pm 0.1} \text{Er}_{0.36 \pm 0.02} \text{F}_{2 \pm 0.5} \text{O}_y$. The presence of oxygen (y) is difficult to quantify with the RBS technique but evident. In the case of a stoichiometric synthesis, it is well known that a solid solution of CaF₂ and ErF₃ presents a unique and very stable crystallographic phase until $(0.4 \text{ErF}_3 + 0.6 \text{CaF}_2)$ ¹⁹. The chemical reaction involved is the following:



Actually, the ratio $\text{Er}/\text{Ca}=0.36/0.7=0.5 \pm 0.1$ is in good agreement with (1), indicating that the major fraction of the erbium ions ($80\% \pm 20\%$) is in a chemically stable state. However, equation (1) assumes the conservation of electric charge and consequently each Er^{3+} require charge compensation which is normally (in the stoichiometric case) fixed by an interstitial F^- ion²⁰. In the implantation process, there is a lack of anions. This first observation leads us to examine further the role of oxygen.

Figure 3 presents the Secondary Ion Mass Spectroscopy (SIMS) profiles of an Er-implanted CaF₂/Si thin film before (Fig. 3a) and after annealing in air at 800°C for one hour (Fig. 3b). Note that the intensities reported in the SIMS figures are not related to the element concentration. Only the shapes of the profiles are discussed. For the as-implanted sample, one can see the effect of the implantation damage and recoil processes on the fluorine profile. Numerous F^- ions were "pushed" into the bulk of the CaF₂ film. The erbium concentration profile is gaussian with a tail that stops before the interface CaF₂/Si (there is no erbium in Si). One can see the adsorption of oxygen at the surface of the sample, due to the moisture in the air. This point can reveal the hygroscopic nature of the fluoride thin film. Indeed, a previous study has shown the possibility of O-H groups adsorption for non-stoichiometric CaF₂/Si thin films¹⁴. After annealing, the SIMS-F profile is flat again. This is certainly related to the fact that we heal the implantation damages due to recoil effects. As can be seen on the SIMS-Er profile, the concentration of Er in the bulk of the film increases due to a diffusion of the implanted Er ions.

An accumulation is observed at the interface between the CaF₂ thin film and the Si substrate. There is no diffusion of Er into the silicon substrate. This diffusion could be substitutional, as demonstrated by Ch. Buchal et al.²¹ for Er-implanted LiNbO₃. As these authors described for Er-implanted LiNbO₃, one can observe in Er-implanted CaF₂ that the Er peak concentration moves slightly to the surface. Most of the Er ions in this region are supposed to be in interstitial positions. One of the most interesting things, however, is to look at the SIMS-oxygen profile after annealing (Fig. 3b): this O-profile fits exactly the erbium one. The oxygen adsorbed at the surface has diffused to the Er-implanted region. This points out the role of oxygen in the charge compensation mechanism that can not be realized by supplementary interstitial F⁻ ions.

Unfortunately, we have not been able to estimate the O content because of the difficulty of quantitative SIMS analysis. Additional investigations are in progress and should help us to confirm the existence of Er-O or Ca-O bindings. Nevertheless, one can suppose the formation of an oxifluoride compound with a formula approaching: Ca_{1-x}Er_xF₂O_{x/2}.

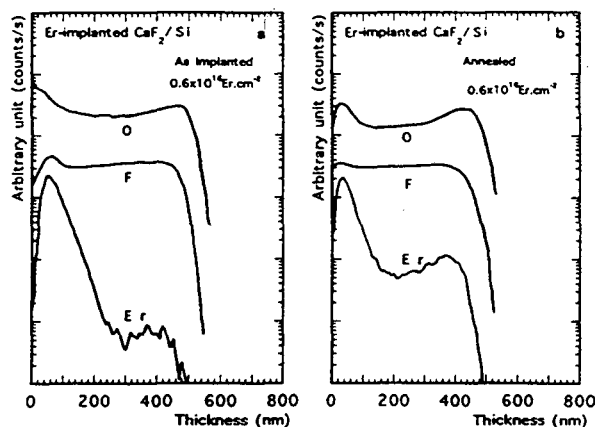


Figure 3 - SIMS profiles of Er-implanted CaF₂/Si structures ($0.6 \times 10^{16} \text{cm}^{-2}$ - 150keV)
 a: Before annealing
 b: After annealing, 800°C, 1h.

PHOTOLUMINESCENCE PROPERTIES

As we discussed in the introduction, the emission of light by erbium ions is due to intra- $4f^{11}$ electronic transitions. This partially-filled shell is well screened by outer closed $5s^2$ and $5p^6$ orbitals, explaining the extreme energetic stability of PL emission lines versus temperature in host material or other “external” conditions. Therefore, as all the excited states of Er³⁺ ions are energetically aligned, very strong resonant effects are observed due to dipole-dipole interactions between two or more Er³⁺ ions.

One of the first limits in the luminescence is the well known concentration quenching phenomenon that occurs for high concentration of luminescent centers. This quenching can be seen as a matrix-coupling effect which results from a multiphononic relaxation and absorption of the initial Er-energy by the matrix. This could be an explanation of the very low quenching-concentration usually observed for Er-doped semiconductors²². In this last case a strong coupling has been observed between the rare earth and the matrix^{23,24}. However, in the case of Er-doped insulating materials, this RE-matrix coupling effect (concentration quenching) is far more limited. Due to larger band gaps (10.4 eV for CaF₂ thin films) and lower phonon energies, the radiative efficiencies are very high in this kind of material.

When included in a fluoride matrix, the erbium ions can be excited by the emission lines of an Argon ion laser at $\lambda=488\text{nm}$ or $\lambda=514.5\text{nm}$ by direct absorption respectively on the $^4F_{7/2}$ and $^2H_{11/2}$ levels of erbium. Thus, we have demonstrated^{12,25,26} that the energy decay from the initially-excited state to the $^4I_{13/2}$ level (1.53 μm) is due to several energy transfers between and within Er ions. These phenomena known as “cross-relaxations” are extremely enhanced in fluoride matrixes^{27,28}. Let us say that in the case of Er-doped CaF₂, the “macroscopic” effect of the cross relaxation mechanisms is to depopulate the high energy emitting levels (green and red), to the benefit of the IR one (1.53 μm)¹². This is the explanation why a very high concentration of active centers can be incorporated in fluoride crystals before attaining the concentration quenching limit. Another effect of energy coupling between or within rare earth ions is the upconversion process^{29,30} which results from a multi-photon absorption or from energy transfer and photon addition between coupled-Er³⁺ ions. We will see that all of these energy-transfer phenomena are observable in the case of Er-implanted CaF₂/Si thin films.

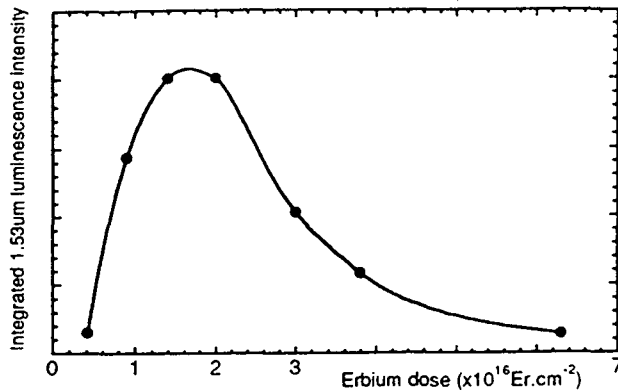


Figure 4 - Evolution of the $^4I_{13/2}$ - $^4I_{15/2}$ luminescence signal at $1.53\mu\text{m}$ as a function of Er implanted dose. excitation: 488nm - 250mW

phenomenon becoming greater. This optimum concentration of $2 \times 10^{16} \text{at.cm}^{-2}$ corresponds to a peak-concentration of $4.5\text{at}\% \text{Er}$, i.e. $x=14\%$ using Equ. (1). This result is three orders of magnitude greater than in the case of Er-doped semiconductors, and two orders of magnitude greater than in the case of Er-doped optical fibers amplifiers. This is to compare with the same optimal concentration measured in the case of MBE-grown $\text{Ca}_{1-x}\text{Er}_x\text{F}_{2+x}/\text{Si}$ samples ($x=16\%$)¹². This slight difference could be due to an imperfect healing of the structural defects after annealing, to an incomplete charge compensation, or to the presence of oxygen. Further investigations have to be done in order to draw a more definite conclusion. Dealing with optical activity, the implantation process reveals a great potential for incorporating erbium in high-active sites. When one compares the luminescence intensity at optimum concentration, of an MBE-grown $\text{Ca}_{1-x}\text{Er}_x\text{F}_{2+x}/\text{Si}$ sample with Er-implanted CaF_2/Si , a factor of roughly two swing the balance in the implanted-sample's favor.

In addition, the Er-implanted samples show a strong up-conversion efficiency. Figure 5 shows the $^4S_{3/2}$ level emission in the green range after pumping the IR- $^4I_{11/2}$ -level at 980nm (100mW) with a GaInAs laser diode. The involved upconversion process is schematized in the insert. The CaF_2/Si sample was implanted to a dose of $0.6 \times 10^{16} \text{Er.cm}^{-2}$.

Finally, we would like to report some low-temperature photoluminescence measurements (LTPL). By freezing the sample at a temperature of 6K , one can see the effect of the crystal field splitting of the Stark sublevels. This weak interaction between the surrounding atoms and there position respectively to the Er ions produces a degeneration of all the $4f^{11}$ levels. Each particular crystallographic site will produce a particular degeneration and a specific energetic scheme. A similar effect will be observed when modifying the nature of the neighboring atoms (Er-F, Er-O, Er-Er, ...). Figure 6 shows the LTPL ($T=6\text{K}$) spectrum of CaF_2/Si implanted structures (a: $0.8 \times 10^{16} \text{Er.cm}^{-2}$), (b: $2 \times 10^{16} \text{Er.cm}^{-2}$). We compare these spectra with the room temperature PL (RTPL) signal (c: $2 \times 10^{16} \text{Er.cm}^{-2}$). At low concentration and at $T=6\text{K}$, a particular site (called A) seems to be dominant. This site is certainly of high symmetry, due to the extreme narrowness of the peaks ($\approx 20\text{\AA}$). When increasing the concentration of erbium (spectrum b), the response of the A site becomes less important and a new set of lines appears (called B). These B site never stop growing as one increases the Erbium concentration. The appearance of the B site could be related to lower symmetric or chemical environment involving stronger $4f$ -shell oscillator strength. In fact, the B site seem to be more active than the A site at room temperature, as indicated by the arrows on figure 6.

Figure 4 shows integrated luminescence intensity at room temperature for the $1.53\mu\text{m}$ radiation versus implantation dose. The samples were annealed at $T=500^\circ\text{C}$, for 1h in air ($20.95\% \text{O}_2$ content). This annealing procedure has been found to be the most efficient one to increase the luminescence efficiency. Each experimental point corresponds to the integrated photoluminescence signal. For excitation, we used the 488nm line of an Argon ion laser (250mW). For low Er-implanted doses, the PL signal rise up due to the increase of active centers and the cross relaxation effects which multiply as erbium concentration extends and Er-Er distances decreases. For implantation doses greater than $2 \times 10^{16} \text{at.cm}^{-2}$, the PL intensity declines, the concentration quenching

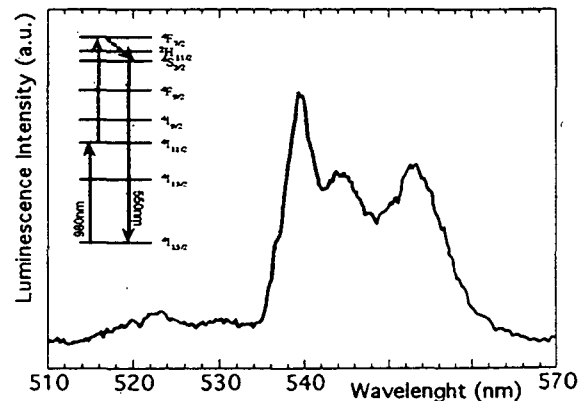


Figure 5 - Green upconversion luminescence of the $^4S_{3/2}$ level of erbium under excitation in the infrared at 980nm - 100mW

CONCLUSION

We have studied the implantation mechanisms of erbium in CaF_2/Si epitaxial structures. We have shown that sputtering processes produce a limitation of the projected range ($150\text{\AA} < R_p < 400\text{\AA}$) at an implantation energy of 150 keV. A study of the annealing conditions in air has shown the dominant role of oxygen diffusion in the charge compensation mechanisms. Annealing in air (500°C , 1h) is necessary to obtain good luminescence efficiencies. The optical properties of the material are of great interest. We obtain strong luminescence intensities at room temperature and a very high optimum concentration of active centers can be reached without quenching of the $1.53\mu\text{m}$ emission. A study of the degeneracy of the $^4I_{15/2}$ level indicates two different types of erbium sites. These excellent optical properties make Er-implanted CaF_2/Si structures a good candidate for the fabrication of active planar waveguides.

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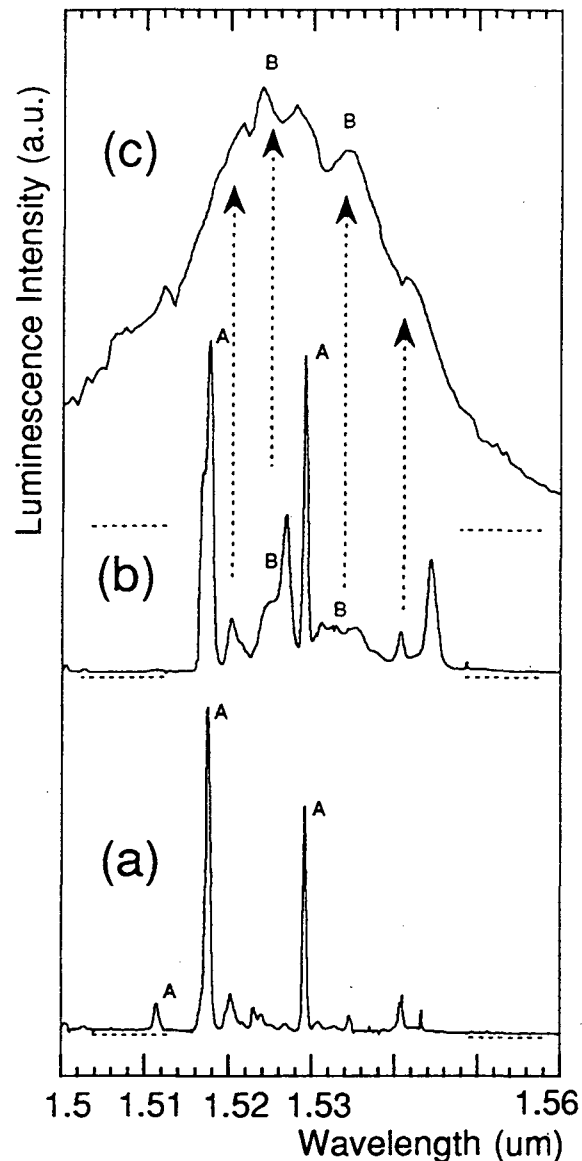


Figure 6 - Luminescence spectra at $1.53\mu\text{m}$ of Er-implanted CaF_2/Si structures.

(a) $T=6\text{K} - 0.8 \times 10^{16} \text{at.cm}^{-2}$

(b) $T=6\text{K} - 2 \times 10^{16} \text{at.cm}^{-2}$

(c) Room Temperature - $2 \times 10^{16} \text{at.cm}^{-2}$

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