1.3-μm Emission of Nd : LaF_3 Thin Films Grown by Molecular Beam Epitaxy

X. Zhang, F. Lahoz, C. Serrano, G. Lacoste, and E. Daran

***Abstract*—The 1.3-μm emission of Nd^{3+}-doped LaF_3 thin films grown on LaF_3 and CaF_2 (111) substrates by molecular beam epitaxy is reported. The waveguide behavior of the heteroepitaxial layers has been demonstrated and the refractive indexes measured. Guided spectra have been obtained from these layers using a prism-coupling technique. The 1.3-µm emission corresponding to the $^4F_{3/2}\rightarrow{}^4I_{3/2}$ transition has been characterized as a function of Nd^{3+} concentration and temperature. The relative efficiencies of different excitation bands were compared. The optimum concentration for Nd^{3+} dopant has been found to be about 1 at.%. A narrowing of the emission lines is observed in the homoepitaxial layers compared to the heteroepitaxial layers. The decay of the luminescence of the $^4F_{3/2}$ level measured at room temperature is similar for homoepitaxial and heteroepitaxial layers.**

***Index Terms*—1.3-μm emission, LaF_3, molecular beam epitaxy, Nd^{3+}.**

I. Introduction

THERE EXIST two intrinsic low-loss telecommunication windows in silica-based optical fiber communication systems: one is at 1.55 µm and the other is at about 1.31 µm. The development of the erbium-doped fiber amplifier (EDFA) operating at 1.5 µm during the last decade has already brought a tremendous increase in optical fiber communication capacity. However, a large component of the worldwide terrestrial telecommunications network operates at the second low-loss window at 1.28–1.32 µm [1]. Unfortunately, the performance of the optical fiber amplifier and laser operating at this wavelength is far less efficient than those operating at 1.5 µm, and the high gain coefficient of 0.36 dB/mW demonstrated at 1.3 µm with a Pr : PbF_2/InF_3-glass amplifier is far smaller than the high EDFA value of ~11 dB/mW [2]. Therefore, much research has been undertaken to develop efficient optical systems operating in the 1.3-µm window.

The possibility of using rare-earth (RE) ion doped systems to meet the requirement of the 1.3-µm telecommunication has been studied for a long time. According to their energy level diagrams, three RE ions have been chosen as possible candidates: Pr^{3+} [3], Nd^{3+} [4], and Dy^{3+} [5] ions. The Nd^{3+} ion was the first to be investigated for this purpose. Although it usually gives rise to an emission ($^4F_{3/2}\rightarrow{}^4I_{13/2}$) on the long-wavelength side of the 1.3-µm telecommunications window and sometimes encounters a serious problem of excited state absorption (ESA) in this wavelength range, its high luminescence efficiency is attractive to researchers. In fact, laser emissions from the 1.3-µm transition have been obtained in various fluoride and oxide crystals such as LaF_3, YAG, and $LiNbO_3$ at both 77 K and at room temperature [6]. In LaF_3 bulk crystals, laser emission in the 1.3-µm band range has been observed by Kaminskii *et al.* [7] at 300 and 77 K with a very low threshold. As for optical amplification, a 10-dB gain has been reported by Sugama *et al.* in a ZBLAN glass-based fiber amplifier [3]. Even higher gain has been predicted in this material by Zemon *et al.* [8]. Moreover, the refractive index of these fluoride materials is a good match to silica-based optical fibers, leading to low reflection losses, and hence a good waveguide-to-fiber coupling efficiency. All these results indicate that Nd^{3+}-doped fluoride materials are good candidates for this application, due in part to their low phonon energy, which reduces the nonradiative relaxation processes of the excited RE ions increasing the lifetime of the emitting level [9].

Compact and efficient waveguide lasers offer some advantages for applications in integrated optical devices. Due to the small areas in which the light is constrained to be guided, a high-power density of the excitation light is achieved and lower pump thresholds than in bulk samples is expected in the waveguides [10]. Molecular beam epitaxy (MBE) has been demonstrated to be a suitable technique to grow fluoride layers on fluoride or semiconductor substrates and is characterized by easy control of the dopant content and high-quality crystal layers [11]. Laser action at 1.06 µm has recently been shown at room temperature (RT) in Nd^{3+}–doped LaF_3–CaF_2(111) MBE grown planar waveguides [12]. This result strongly supports the idea that these systems can be seriously considered as a candidate for RT 1.3-µm lasers.

In this paper, we report the ordinary and guided 1.3-µm emission in MBE grown Nd^{3+}-doped LaF_3–CaF_2(111) heteroepitaxial planar waveguides, as well as refractive index results. The luminescence properties of Nd^{3+}-doped LaF_3 homoepitaxial layers grown by MBE are also studied, and the results are compared and discussed in order to improve these systems for laser or amplifier applications.

II. Experimental

Nd^{3+}-doped LaF_3 layers have been grown by MBE using two different effusion cells for LaF_3 and NdF_3, which allows the control of the composition of the thin films. A polished

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TABLE I
REFRACTIVE INDEX VALUES FOR THE TE AND TM MODES MEASURED AT DIFFERENT WAVELENGTHS IN A LaF_3 LAYER GROWN ON A CaF_2 (111) SUBSTRATE

λ (nm)	**Refractive index (n)**	
	TE modes ($n_{ordinary}$)	TM modes ($n_{extraordinary}$)
488	1.6097 ±2.10^{-4}	1.6026 ±2.10^{-4}
514	1.6082 ±2.10^{-4}	1.6000 ±2.10^{-4}
633	1.6033 ±2.10^{-4}	1.5953 ±2.10^{-4}

LaF_3 crystal was used as a substrate for the homoepitaxial layer and commercially available polished CaF_2(111) crystals for the heteroepitaxial layer. The nominal concentrations determined from the calibration curves for NdF_3 dopant range from 0.5 to 5 at.%. Undoped LaF_3–CaF_2(111) layers were also prepared. Epitaxial growth was performed at a substrate temperature of about 520 °C and a typical growth rate of 0.7 μm/h. The layer thickness of the obtained films is about 3.6 µm. The lattice parameter mismatch between the $Z = 2$ subcell of the hexagonal basal plane of LaF_3 and the hexagonal symmetry unit of the CaF_2(111) surface is about 7% and could result in strains and/or disorder in the layers. Nevertheless, the thin films were found to be free of cracks and exhibited a featureless surface under optical microscopy.

A photoluminescence study was performed by using a tunable Ti : sapphire CW laser as the excitation source. The sample was fixed in a cryostat circulated by liquid helium, and the temperature can be continuously changed from 25 K to room temperature. The luminescence signal was dispersed by a 1-m monochromator and detected by a liquid-nitrogen cooled Ge detector. The guided spectra for the active layers were obtained using a prism-coupling technique. The emission signal from the guide was collected by a cylindrical lens and focussed onto the entrance of the monochromator. For luminescence decay measurements, the excitation beam was modulated with a mechanical chopper. The decay signal detected by a photomultiplier (PM 636) was recorded on a digital oscilloscope (Philips PM/3323).

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Refractive Index

The refractive index n of the heteroepitaxial layers was measured by the m-lines method as a function of the wavelength and the dopant concentration. The optical quality and flatness of the surface layer was confirmed by the good optical coupling achieved when a glass prism was placed over the film fixed with pressure. Doping with Nd^{3+} results in a hardly detectable modification of the indexes for the studied concentration range (0.5–5 at.%). For the undoped LaF_3–CaF_2(111) layer, several guided modes were observed at 488, 514.5, and 633 nm, and a small birefringence was also detected. Table I shows the measured ordinary and extraordinary refractive indexes. The same values are found as those observed for LaF_3 bulk crystals at the wavelengths studied [13]. It therefore seems reasonable that their wavelength dependence can also be applied for the MBE-grown films. With this assumption, calculations show that four modes are guided in the studied heteroepitaxial layers at 1.3 µm. It is necessary to reduce the thickness of the layer to about 1 µm to obtain a monomode guide, which results in a practical problem of coupling the pump light into the end-face of the guide. On the other hand, the large difference between the refractive index of the CaF_2 substrate and the LaF_3 active layer gives rise to a high numerical aperture of about 0.71 at 790 nm, which is favorable for an efficient direct coupling of a diode laser into the end-face of the planar waveguide.

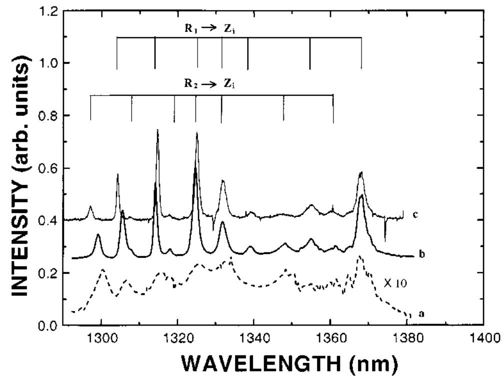

Fig. 1. Emission spectra of the Nd^{3+} $^4F_{3/2} \rightarrow {^4I_{13/2}}$ transition in (a) LaF_3: 1 at.% Nd^{3+}–CaF_2(111) at 300K, (b) LaF_3 :1 at.% Nd^{3+}–CaF_2(111) at 25K, and (c) LaF_3 :1 at.% Nd^{3+}–LaF_3 at 25K . The bar diagrams show the transitions between the Stark sublevels of the $^4F_{3/2}$ and the $^4I_{13/2}$ manifolds.

For the homoepitaxial sample, both the layer and substrate have the same refractive index and the light cannot be guided into the film. However, it has been observed that codoping with certain elements significantly increases the n value of the media [14]. In the case of LaF_3, calculations show that a small relative increase of $\Delta n = 0.19\%$ leads to single-mode operation at 1.3 µm in a 4-µm-thick waveguide. Low concentration codoping with some RE such as CeF_3 [13] could probably be enough to produce this effect.

B. Luminescence Properties

A typical emission spectrum of the heteroepitaxial layers is shown in Fig. 1(a) and (b) at room temperature and 25 K, respectively, exciting at about 788 nm. The observed emission lines have energies slightly different from those reported for bulk crystals [15], [16]. Two partially overlapped patterns of seven lines corresponding to the transition from the two Stark sublevels of the $^4F_{3/2}$ level (R_i) to the seven Stark sublevels of the $^4I_{13/2}$ manifold (Z_i) are observed in the low temperature spectrum (see the bar diagrams in Fig. 1). With increasing temperature, the emission intensity is decreasing progressively, and, at room temperature, the integrated emission intensity decreases to about 1/5 of that at 25 K. Two reasons can be responsible for this diminution: the first is the increase of the nonradiative transition probability with increasing temperature, and the second is the redistribution with temperature of the branching ratio of the $^4F_{3/2}$ to the different ^4I_j levels. We have noticed that the infrared emission located between 840–900 nm corresponding to the $^4F_{3/2} \rightarrow {^4I_{9/2}}$ transition is reinforced with increasing temperature, which means a decrease in the branching ratio to the other ^4I_j levels.

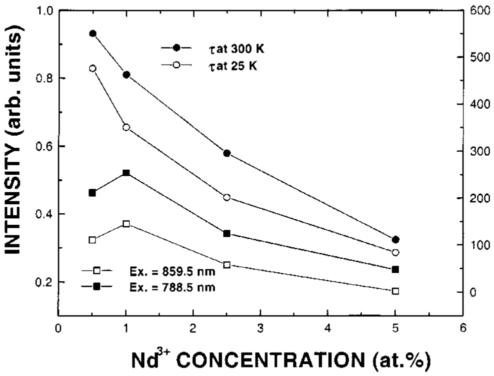

Fig. 2. Relative intensity of the 1323-nm emission line measured at 25 K under two excitation wavelengths as a function of Nd^{3+} concentrations for the heteroepitaxial layers. Decay times at 25 and 300 K of the $^4F_{3/2}$ level of Nd^{3+} as a function of the Nd^{3+} concentration in these layers and in LaF_3 : 1 at.% Nd–LaF_3 are also shown.

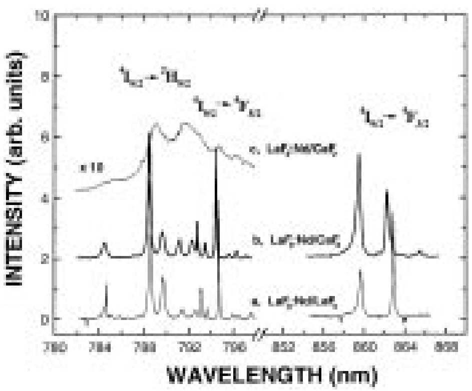

Fig. 3. Excitation spectra for the monitoring emission line at 1323 nm of Nd^{3+} ions in LaF_3 layers grown on LaF_3 substrates at (a) 25 K, (b) on CaF_2 (111) at 25 K, and (c) at room temperature.

The emission spectrum of the LaF_3 : Nd—LaF_3 layer measured at 25 K under 788.5-nm excitation is shown in Fig. 1(c). Slight differences in the positions of the lines compared with those of bulk samples are observed. A narrowing of the linewidth is seen between the homoepitaxial and the heteroepitaxial layers. The strains, defects, and/or disorder of the LaF_3 films, which originated from the lattice mismatch and thermal expansion differences between the layer and the substrate, are avoided in the homoepitaxial growth and the narrowing of the lines suggests that a better crystal quality is achieved. It is important to note that the crystal quality of the films is an important factor in the propagation losses obtained in the waveguide.

The dependence of the emission intensity on the Nd^{3+} concentration has been studied in the heteroepitaxial layers. The results are shown in Fig. 2. The most intense emission is observed for the 1 at.% layer both under 788.5- and 859.5-nm excitations. For higher Nd^{3+} concentration, quenching of the luminescence occurs, which is in agreement with the results observed in bulk crystals [17].

The excitation spectrum for 1% Nd^{3+} doped layers is given in Fig. 3 for both heteroepitaxial and homoepitaxial samples. These spectra can be ascribed to three transitions of Nd^{3+} : $^4I_{9/2} \rightarrow {}^4F_{3/2}$ (852–864 nm), $^4I_{9/2} \rightarrow {}^4F_{5/2}$(792.5–798 nm) and $^4I_{9/2} \rightarrow {}^2H_{9/2}$(780–792.5 nm). According to the relative peak intensities, the excitation efficiency for the $^4I_{9/2} \rightarrow {}^2H_{9/2}$ transition at 788.5 nm is higher than that of the $^4I_{9/2} \rightarrow {}^4F_{3/2}$ transition at 859.5 nm, for all the concentrations studied. The energy levels of the three manifolds involved in the excitation spectra can be deduced from Fig. 3 and the results are shown in Table II. Values from bulk LaF_3 : Nd crystals reported earlier by Caspers *et al.* [15] and Vignanesvara *et al.* [16] are also collected in Table II for comparison. Small energy differences are observed between the layers and the bulk crystal. Notice that the energy interval between the two crystal field-splitting components of the $^4F_{3/2}$ level is 37 cm^{-1} for LaF_3 : Nd—CaF_2 (111), while those for the homoepitaxial layer and bulk crystal are both about 42 cm^{-1}. The difference is probably due to the lattice mismatch and the difference in expansion coefficient between the layer and the substrate, which results in the strain and defects in the epitaxial layers.

At room temperature, the short-wavelength excitation spectrum becomes a broad band ranging from 782 to 798 nm centered at about 792 nm [Fig. 3(c)]. This band covers most of the emission regions of commercially available high-power diode lasers.

Guided spectroscopy was performed at room temperature, taking advantage of the waveguiding behavior of the heteroepitaxial samples. In this configuration, excitation light is coupled into the doped layer by a glass prism. The main advantage of this configuration is a huge enhancement of the emission intensity, which is estimated to be at least an order of magnitude higher than in the ordinary nonguided configuration. The obtained guided spectrum in the spectral region of 1.3 μm at room temperature has the same shape and line positions as that of the nonguided configuration (see Fig. 1(a)). The only difference is a large increase in the luminescence intensity.

It is worth mentioning that, for the room-temperature guided spectra, the integrated emission intensity for the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition (at 1.06 μm) is about 4.8 times of that of the $^4F_{3/2} \rightarrow {}^4I_{13/2}$ transition (at 1.3 μm). This value is close to the proportion of the theoretical branch ratios (about 4.6) [18] of these two transitions calculated by the Judd–Ofelt theory from the room—temperature absorption spectra of Nd^{3+} in LaF_3 bulk crystals. Obviously, for the theoretical results, the excited state absorption (ESA) was not taken into account. As reported by Page [5], in some materials, like oxide glasses, ESA is very important, completely overwhelming the stimulated-emission gain, and actually leading to pump-induced loss at 1.3 μm. The results observed in this work indicate that, in the LaF_3 films, the ESA of Nd^{3+} at 1.3 μm is probably not an efficient process.

It is important to note that in a planar waveguide the light is confined in just one direction. An additional confinement in another direction can be achieved in channel waveguides, typically leading to an order of magnitude increase in the gain. Therefore, as laser action at 1.06 μm has already been demonstrated in LaF_3 : Nd planar waveguides at room temperature, it can be expected that, even if the branching ratio from $^4F_{3/2}$ to $^4I_{13/2}$ is lower than that to $^4I_{11/2}$, laser action at 1.3 μm could be obtained in channel waveguides. Channel waveguide fabrication and laser tests at 1.3 μm will be shortly attempted in both heteroepitaxial and homoepitaxial MBE samples.

TABLE II
THE $^4F_{3/2}$, $^4F_{5/2}$, AND $^2H_{9/2}$ ENERGY LEVEL POSITIONS OF Nd^{3+} MEASURED IN LaF_3 EPITAXIAL LAYERS AT 25 K (ENERGY IN AIR). VALUES FOR BULK CRYSTALS ARE ALSO PRESENTED

SLJ manifold	Values of this work (cm^{-1})		Values in bulk crystal (cm^{-1})	
	Homoepitaxy	Heteroepitaxy	Ref[15]	Ref[16]
$^4F_{3/2}$	11590 11632	11597 11634	11595 11637	11592 11634
$^4F_{5/2}$	12584 12601 12609	12588 12603 12613	12596 12613 12621	12596 12615 12622
$^2H_{9/2}$	12663 12682 12745	12664 12682 12747 12841	12675 12693 12755	12676 12694 12753 12842 12904

C. Lifetime Measurements

Another important spectroscopic parameter to characterize the luminescent behavior of Nd^{3+} ions in these films is the decay time of the emitting level. The results measured at both 25 and 300 K for a series of LaF_3–Nd layers with Nd^{3+} concentrations from 0.5 to 5 at.% are shown in Fig. 2. These values are obtained by monitoring the Nd^{3+} $^4F_{3/2}\rightarrow{}^4I_{9/2}$ transition at about 860 nm. A nonexponential decay curve was observed for all the concentrations. The criterion chosen to evaluate the decay time is the $1/e$ dropping time. As shown in Fig. 2, for all the samples, the 300 K decay times are always longer than those observed at 25 K. This unusual temperature dependence feature has also been observed for Nd^{3+} -doped LaF_3 bulk crystals and has been interpreted by Voron'ko *et al.* [17] as the effect of the energy migration among the Nd^{3+} ions, and the energy transfer to inactive centers. They have shown by experimental results and model calculations that the energy migration probability decreases as the temperature goes beyond 50 K. This results in the increase of the luminescence decay times with increasing temperature. It is difficult to compare decay time values with those given in the literature as the calculation criterion is not always known. The values for other systems are given for information. The values for 1 at.% Nd^{3+}-doped samples are 470 µs in $LaF_3:Nd–CaF_2$, 470 µs in $LaF_3:Nd–LaF_3$, and, in the literature, 516 µs in $LaF_3:Nd–Si$, 655 µs in $LaF_3:Nd$ bulk crystal [19] and 240 µs for Nd^{3+}-doped YAG. It appears that no lifetime shortening is taking place in the heteroepitaxial waveguides with our calculation criterion.

IV. CONCLUSION

Nd^{3+}-doped LaF_3 heteroepitaxial planar waveguides grown on CaF_2 (111) substrates by MBE have been characterized. The same refractive index as that found in bulk samples is obtained. Efficient 1.3-µm emission has been detected and investigated as a function of Nd^{3+} doping concentration at both 25 K and at room temperature, and guided spectra were obtained by using a prism-coupling technique at room temperature. The optimum Nd^{3+} concentration for luminescence intensity is about 1 at.%. The most efficient excitation band is observed for the $^4I_{9/2}\rightarrow{}^2H_{9/2},{}^4F_{5/2}$ transitions located at room temperature between 780 and 800 nm, which is compatible with commercially available high-power diode lasers [20]. A narrowing of the emission lines is observed in homoepitaxial samples, suggesting an improvement of the crystal quality. In this case, codoping with an additional impurity is necessary to increase the refractive index of the layer. As value of Δn as small as 0.19% would be enough to obtain monomode operation at 1.3 µm in a 4-µm-thick homoepitaxial waveguide. The conditions for MBE growth of low-loss, high-crystal-quality, thin film waveguides is under study.

The fact that laser action at 1.06 µm has been obtained by the authors in $LaF_3:Nd^{3+}$ planar waveguides with less than 1 dB/cm of propagation loss and lasing threshold of 80 mW [12] and that 1.3-µm laser emission has already been already obtained in $LaF_3:Nd$ bulk crystals, both at 300 and 77 K suggests that laser action at 1.3 µm can be envisaged in $LaF_3:Nd^{3+}$ channel waveguides, for which a higher confinement of light is achieved. Nevertheless, absorption and stimulated emission cross sections measurements should be performed to determine the suitability of $LaF_3:Nd^{3+}$ thin films for laser operation.

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