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Optical response and surface morphology of crystalline Nd^{3+}-doped fluoride films grown on monocrystalline $LiYF_4$ substrates by pulsed laser deposition

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ABSTRACT

The realization of crystalline films of Nd^{3+}:YF_3 and Nd^{3+}:$LiYF_4$ on a monocrystalline $LiYF_4$ substrate by pulsed laser deposition is reported. The films were obtained by laser ablation with 355 nm photons of a bulk $LiYF_4$ crystal doped with Nd^{3+} ions at 1.5% atomic concentration in the presence of different ablation/deposition parameters. The films optical characteristics, analyzed via laser induced polarized fluorescence spectroscopy upon IR excitation, are presented. Lifetime measurements of the fundamental Nd^{3+} ion transition in the film were also performed. All these results were compared with those obtained in the Nd^{3+}:$LiYF_4$ bulk crystal. The surface morphology of the depositions was analyzed via a scanning electron microscope. When the production of the deposition took place in high vacuum (1×10^{-4} Pa) and the substrate temperature was 750 °C, the grown film was Nd^{3+}:YF_3. A 1 Pa controlled atmosphere of He in the ablation chamber and a substrate temperature of 650 °C favoured the growth of a Nd^{3+}:$LiYF_4$ film. In the latter case the film showed also a smoother surface.

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1. Introduction

The outstanding qualities of fluoride crystals as hosts for rare-earth (RE) ions to produce emission extending from IR to UV are well known. In particular they provide remarkable laser sources in the IR region when compared with RE-doped oxides as they have lower phonon energies and comparable emission cross sections [1]. A significant improvement of the optical properties of the RE-doped crystals as laser materials can be achieved via their production in a film shape. This has already been proven true for films of a large number of RE-doped oxides [2–4] and it is the reason why, in spite of the severe technical difficulties, there has been in recent years a growing effort in the attempt to produce RE-doped fluoride crystals in a film shape. The first results were obtained by liquid phase epitaxy [5], vacuum deposition and sol–gel technique [6] and pulsed laser deposition (PLD) mainly on amorphous or Si substrates [7–9]. The technique that comes forth as the most versatile and appropriate is PLD essentially because it permits the congruent transfer from the bulk to the film and the possibility of operating both in high vacuum or in a background gas controlled atmosphere. We introduced a new aspect in this research by using the same undoped monocrystalline bulk fluoride crystal as the substrate for the PLD film growth [10]. This situation guarantees a good accordance for the crystal lattice constants in the interface between the film and the substrate so facilitating the development of real crystalline depositions. The very first result obtained by laser ablating a Nd^{3+}:$LiYF_4$ (Nd:YLF) crystal with subsequent deposition on a monocrystalline $LiYF_4$ (YLF) substrate was a Nd^{3+}:YF_3 (Nd:YF) film with rather poor optical properties and surface quality, but already showing a crystalline nature [10]. In the following we report about the PLD production of sub-micrometric crystalline Nd:YF and Nd:YLF films on a pure monocrystalline YLF substrate obtained by ablating with 355 nm laser pulses a monocrystalline 1.5% atomic concentration (at.) Nd:YLF crystal in different experimental conditions. The optical response of the films was analyzed and the lifetime measurements of the fundamental Nd^{3+} ion transition in the film were performed, while the quality of the films surface was checked by a scanning electron microscope (SEM). It will be shown that a lower temperature of the substrate and the presence of a 1 Pa controlled He atmosphere in the ablation chamber favoured the production of Nd:YLF films of good quality, with a Nd^{3+}-ion concentration very close to that of the bulk and a surface morphology better than that of the Nd:YF film.

2. Experimental details

The whole experimental apparatus is described in ref. [10] and in the utmost detail in ref. [11]. Here we recall only those features

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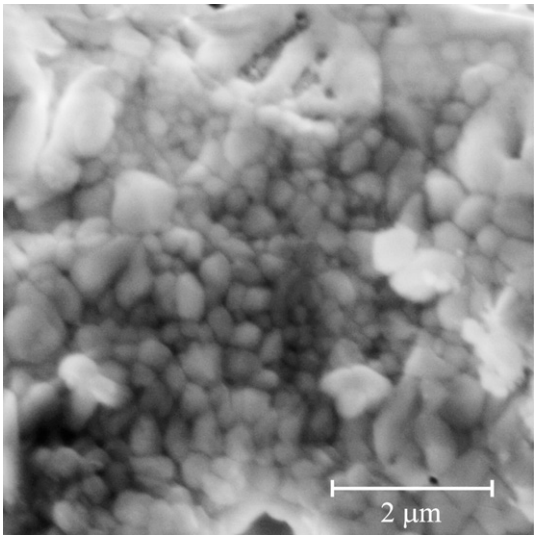

Fig. 1. SEM picture of the Nd:YF film deposited with 10 J/cm^2 laser fluency on the YLF substrate heated to 750 °C in vacuum.

essential for the present experiment. The films were deposited by PLD using 355 nm laser pulses provided by the third harmonic of a Nd-YAG laser at 10 Hz repetition rate. The maximum pulse energy was 130 mJ and its duration was 13 ns. The laser beam, focused to an 800 μm diameter spot, hits the target at 45° to the surface normal and was translated 650 μm horizontally every 7 min. In this way the target provided frequently fresh polished surface to the ablating beam and the stability of the ablated plume axis orientation was favoured. The ablation took place with a laser fluency set at 10 J/cm^2, with 12,000 laser shots after about 1500 target cleaning shots. The 7.2 mm diameter and 3 mm thick cylindrical target was a monocrystalline YLF bulk crystal doped with Nd^{3+} 1.5% at. The substrate was a pure monocrystalline YLF crystal of similar dimensions with the thickness reduced to 2 mm. Both the crystals had been a-cut as parallel flat faces samples. The bulk was fixed on the target holder and kept rotating at a maximum speed of 1.25 rpm so to prevent craterization. The substrate was mounted on the substrate holder/heater, aligned with the target holder, placed at 3.5 cm distance and equipped with a shutter to cover the substrate during the target cleaning laser shots. The heater can reach temperatures up to 1200 °C with ±1 °C stabilization. As a high substrate temperature should favour the smoothness of the deposited film [12], the temperature of the YLF substrates was either 650 °C or 750 °C, values high enough but still far from the YLF melting point (~810 °C) [13]. Inside the ablation chamber there was either a high vacuum or a controlled pressure of inert gas. After the deposition the substrate was cooled down to room temperature at which all the checks on the films were done. In the effort to minimize the effect of the high fluoride crystals thermal shock sensitivity, special care was dedicated in this experiment to the control of the substrate heating/cooling cycles which were programmed in such a way to increase/decrease the substrate temperature at a rate of 1 °C/min, linearly. The effective deposition of a layer on the substrate with an average estimate of its thickness, and the presence of the Nd^{3+} ions in it were checked inside the vacuum chamber via standard interferometric and laser induced fluorescence (LIF) techniques as described in detail in ref. [10,11]. The comparison between the LIF spectrum produced by the film after grazing incidence excitation with the laser beam at 355 nm, properly attenuated, with the corresponding one recorded from the bulk crystal under the same excitation, proved the presence of the Nd^{3+} ions in the layer. The optical characterization of the films was made outside the vacuum chamber by recording the LIF room temperature spectra either unpolarized or polarized with the electric field (E) || or ⊥ to the *c*-axis of the YLF substrate after excitation of the film with a modulated cw diode laser radiation at 806.6 nm focused to a 600 μm diameter spot. The two investigated regions were those around 900 nm and 1050 nm corresponding to the Nd^{3+} ions emission from the $^4F_{3/2}$ manifold to the $^4I_{9/2}$ and $^4I_{11/2}$ manifolds respectively. The excitation wavelength, λ_{exc}, belonged to the fundamental Nd^{3+} ion absorption band corresponding to the manifolds transition $^4I_{9/2} \rightarrow {}^4F_{5/2} + {}^2H_{9/2}$ and was chosen so to maximize the fluorescence emission from the Nd:YLF bulk crystal. The film LIF spectra were dispersed by a 320 mm monochromator with 1.2 nm resolution, detected by a photomultiplier and processed by a lock-in amplifier. An 830 nm long pass filter in front of the monochromator minimized the scattered excited laser light. For the $^4F_{3/2}$ manifold lifetime measurements, we fitted the decay signal of one of the fluorescence lines belonging to the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ manifolds transition. We used the same excitation diode laser output, chopped at 100 Hz repetition rate so to give laser pulses with extinction time of less than 5 μs. The laser diode power was lowered down as much as possible so to reduce the non-linear effects to the smallest extent. The time resolved signals were detected via the same monochromator/photomultiplier assembly and recorded by a digital oscilloscope for analysis. The total response time of the system was 20 μs. Both the LIF unpolarized and polarized spectra as well as the lifetime measurements were compared with those obtained from the Nd:YLF bulk crystal under the same experimental conditions. Throughout the data acquisition, the experiment

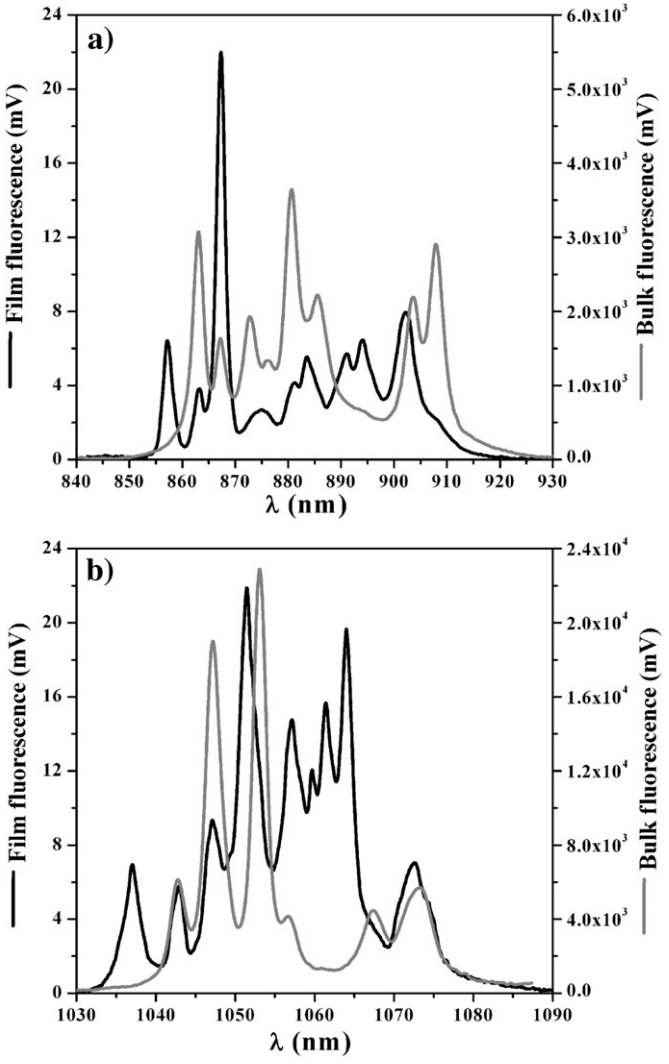

Fig. 2. Comparison between the unpolarized Nd:YLF bulk crystal fluorescence spectra (grey line, right scale) with those of the Nd:YF film (black line, left scale). a) $^4F_{3/2} \rightarrow {}^4I_{9/2}$ manifolds transition; b) $^4F_{3/2} \rightarrow {}^4I_{11/2}$ manifolds transition.

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