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Crystal growth, luminescent and lasing properties of the ytterbium doped $Li_6Y(BO_3)_3$ compound

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Abstract

The luminescence properties of Yb^{3+}-doped $Li_6Y(BO_3)_3$ crystalline material are presented for applications as active medium in diode pumped (short pulse) lasers emitting around 1040 nm and pumped at 975 nm. A single crystal of several centimeter size was grown by the Czochralski method. The absorption and emission spectra were recorded. This borate exhibits a strong absorption line around 975 nm and a broad emission line around 1040 nm. The fluorescence lifetime associated to this intense $^2F_{5/2} \rightarrow {}^2F_{7/2}$ transition is about 1.1 ms. All these parameters make this material a good host for short pulse laser applications. First laser tests have been performed by diode-pumping in a plano-concave cavity.
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1. Introduction

Borate compounds are currently very attractive for the scientific community owing to their wide range of applications (non exhaustive list [1–4]). They exhibit high transparency far in the UV combined with a high damage threshold for exposure to energetic radiations. Our interest in search of new phosphors, laser materials and scintillators led us to the investigation of rare earth doped borate systems. Some of us have earlier underlined the importance of the Ce^{3+}-activated $Li_6Gd(BO_3)_3$ compound as neutron detectors [5].

Recent research on Yb^{3+} doped materials has shown that this rare earth could be a good doping element for efficient diode-pumped short pulse laser materials [6,7]. The simple energy level diagram of Yb^{3+} consists of two manifolds, the $^2F_{7/2}$ ground state and the $^2F_{5/2}$ excited state. As a consequence a high level of ytterbium can be introduced before reaching the critical concentration threshold for luminescence quenching. Since the absorption band in most of the ytterbium materials lies in the range from 900 to 980 nm which is very well covered by the high-power InGaAs laser diodes commercially available, diode pumped ytterbium doped materials are emerging as efficient and compact solid state lasers. Generally, the usefulness of the material for laser applications is evaluated by the fluorescence life time, the emission bandwidth and cross-section as well as the low absorption at the emitting wavelength.

In the present investigation, we report the crystal growth of $Li_6Y_{1-x}Yb_x(BO_3)_3$ ($x = 0.15$) and the spectroscopic properties of the Yb^{3+} ions in $Li_6Y(BO_3)_3$ explored on polycrystalline materials as well as first results of lasing properties in a linear test cavity.

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2. Materials synthesis

2.1. Powder preparation

The synthesis of powders was carried out by a high temperature solid state reaction in oxygen atmosphere from the starting materials Li_2CO_3, H_3BO_3, and Yb^{3+}-doped Y_2O_3 performed from Ln_2O_3 (Ln = Y, Yb) of purity $\geqslant$99.99%. The stoichiometric mixtures of the starting materials ($Yb^{3+}x$%-doped Y_2O_3; x = 1%, 2%, 5%, 10%, 20% 50% and 100% of molar substitution rate) were initially heated at 450 °C for 12 h and then at 750 °C for 15 h, provided with an intermediate grinding.

The purity of the final products was determined by X-ray powder diffraction analysis using CuK_α radiation.

2.2. Crystal growth

The $Li_6Y(BO_3)_3$ phase [8] is isostructural with the $Li_6Yb(BO_3)_3$ phase [9]. It crystallizes in the monoclinic system with the space group $P2_1/c$, $Z = 4$. The structure can be described as made up of chains of edge-sharing YO_8 polyhedra running along the c-axis giving rise to chains. The distance between two successive yttrium atoms in a chain is about 3.85 Å whereas the distances between two yttrium atoms are equal to about 7 and 8.4 Å, along the a-axis and the b-axis, respectively. So the Y–Y interactions along the chains should be predominant over the interchain ones, leading to a monodimensional character.

All borate groups consist of boron triangularly coordinated to oxygen atoms. The lithium atoms occupy six independent positions and are surrounded by four or five oxygen atoms.

The crystal growth of the Yb^{3+}-activated $Li_6Y(BO_3)_3$ has been undertaken by the Czochralski method. A DTA performed on the pure yttrium phase shows that congruent melting occurs at about 860 °C with a high undercooling (150 °C) [5]. The pre-melted product of composition $Li_6Y_{0.8}Yb_{0.2}(BO_3)_3$, was loaded in a platinum crucible of 150 cm^3 and placed in a radio frequency heating pulling apparatus under air atmosphere. The crucible was heated to a temperature slightly above the melting point until all the charge was melted and homogenized. The seed ($Li_6Y(BO_3)_3$ crystal) held on an alumina rod was dipped to touch the melt at a rotation rate of 5–7 rpm. The typical pulling rate was 0.5–0.7 mm/h for a duration of a week to obtain a rod of 15 mm in diameter and of 100 mm length. The crystals are chemically stable and were cut and optically polished using alumina suspension in water.

The diffraction pattern which correspond to the b-oriented crystal is shown in Fig. 1 and compared to the polycrystalline powder obtained from a part of the grown crystal (crushed crystal). All these Bragg peaks can be indexed to a monoclinic unit cell (a = 7.1767(7) Å, b = 16.4205(15) Å, c = 6.6350(6) Å and β = 105.265(5)°) and are in good agreement with the literature values reported for $Li_6Y(BO_3)_3$ [8]. All the observed reflections correspond to the $(0k0)$ planes.

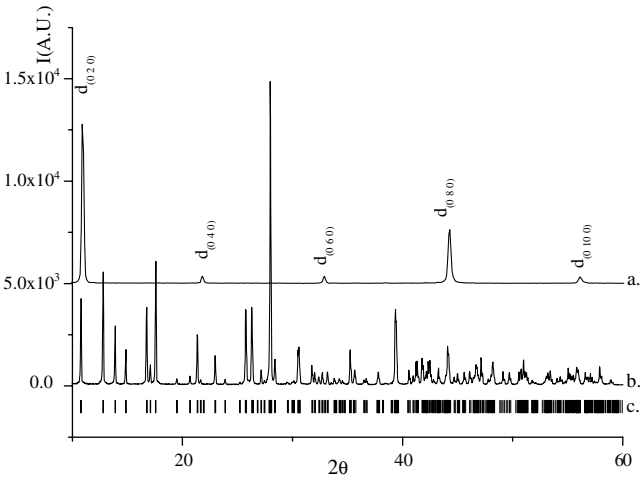

Fig. 1. Superposition of the XRD pattern of the b-oriented single crystal (a), the XRD pattern of the $Li_6Y_{0.85}Yb_{0.15}(BO_3)_3$ composition (crushed crystal (b)) and the theoretical position of Bragg peaks (c).

3. Optical studies

3.1. Experimental setup

3.1.1. Powder characterizations

The emission spectra performed on the powders were recorded using a spectrofluorometer (Edinburgh FL900CDT (M30 monochromator)) connected to a germanium AD 403L detector using a Xe lamp or a laser diode as excitation sources, respectively.

The fluorescence decay curves were recorded by a digital oscilloscope (LeCroy Waverunner LT 342) equipped with a germanium AD 403HS detector coupled to a monochromator CVI R110. The excitation source was a fiber-coupled diode controlled by a fonction generator (HM 8030) delivering a square signal (frequency equal to 4 Hz). The fluorescence lifetimes were recorded on low ytterbium concentration samples to avoid reabsorption and to ensure a single exponential decay.

3.1.2. Single crystal characterizations

The absorption spectra were recorded using a double beam spectrophotometer (CARY UV–VIS–NIR) between 175 and 3300 nm.

The experimental laser setup is presented in Fig. 2. The crystal was placed in a plano-concave cavity pumped by an InGaAs diode, temperature-tuned at 972 nm. The pump beam was first shaped with a telescope and then focused on the crystal by a 76 mm-focal lens. The pump waist in the crystal was a rectangle of

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