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Growth and optical properties of Pr,Yb-codoped KY_3F_{10} fluoride single crystals for up-conversion visible luminescence

Kyoung Jin Kim[a], Anis Jouini[a,*], Akira Yoshikawa[a], Rayko Simura[a], Georges Boulon[b], Tsuguo Fukuda[a]

[a] *Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577, Japan*
[b] *Laboratoire de Physico-Chimie des Matériaux Luminescents, UMR CNRS 5620, Université Claude Bernard Lyon 1, Bat. A. Kastler, 10 Rue Ampère, Villeurbanne 69622, France*

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Abstract

We investigate different ways to realize laser emission from (Pr^{3+}) $^3P_{J=0,1,2}$ levels by pump sources other than the common argon and excimer-dye laser. The use of infrared (IR) laser diodes in combination with intra- and inter-ionic energy transfer processes (up-conversion) could be an efficient solution towards laser oscillation. Pr^{3+},Yb^{3+}-codoped KY_3F_{10} (Pr, Yb:KYF) single crystals were successfully grown by the micro-pulling-down (μ-PD) method. The crystals were transparent with a slightly greenish color, 2.0–2.5 mm in diameter, 20–30 mm in length and free from visible inclusions and cracks. Effective segregation coefficients of Pr and Yb in KYF were studied by means of absorption and chemical analysis. Strong visible emission via selective IR pumping with $\lambda = 975$ nm and up-conversion excitation were obtained in Pr, Yb:KYF at room temperature (RT). Luminescence measurements have been carried out and the decay kinetics of the Pr^{3+} visible emissions was investigated by room temperature time-resolved spectra.
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1. Introduction

The sensitization of rare-earth (RE) doped solid-state laser materials with trivalent ytterbium ions (Yb^{3+}) is a well-known method for increasing the optical pump efficiency by Yb^{3+}-RE energy transfer [1]. The Yb^{3+} ions exhibit a strong and broad absorption band centered at about 980 nm and can be easily pumped by commercially available InGaAs infrared (IR), laser diodes. Moreover, the development of up-conversion lasers as an alternative solution for visible light generation [2] has gained interest in searching for an efficient monochrome IR diode-pumping scheme. One promising technique, which has been demonstrated, is co-doping with Yb^{3+} ions to sensitize up-conversion laser emission at blue, green and various orange-red wavelengths from Pr^{3+} [3–6] and Tm^{3+} [7] in glass fibers. Up-conversion lasing in Yb^{3+} codoped crystals has been reported for a few fluoride systems only, such as Yb:BaY_2F_8 codoped with Ho^{3+} [1], Tm^{3+} [8,9], Er^{3+} [1,9] and Pr^{3+} [10]. Our interest is focused on the trivalent praseodymium ion, which exhibits a variety of laser transitions in the visible range based on 4f–4f transitions from the $^3P_{J=0,1,2}$ level [11,12]. A comprehensive spectroscopic study of Pr:KYF crystals grown by the Bridgman Stockbarger technique was already reported by Wells et al. [13]. Moreover, in Pr, Yb:$YLiF_4$ (YLF) laser oscillation due to Pr^{3+} ($^3P_0$) → Pr^{3+} ($^3F_2$) transition via IR pumping and photo avalanche excitation was realized [11].

Very recently, we have investigated the (Pr, Yb) system in YbF_3–GdF_3 mixed fluoride crystals with respect to realization and better understanding of Pr and Yb energy

*Corresponding author. Fax: +81 22 217 5102.
E-mail address: jouini@tagen.tohoku.ac.jp (A. Jouini).

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transfer and promising results were already reported [14]. The mechanism of up-conversion excitation in Pr^{3+},Yb^{3+}-codoped KYF has never, to our knowledge, been reported. The present work describes the growth and effective segregation coefficients of Pr^{3+} and Yb^{3+} in KYF. Colorless to greenish crack-free single crystals were successfully grown using the micro-pulling-down (μ-PD) method. The absorption coefficient and the up-conversion luminescence in Pr, Yb:KYF single crystals upon selective pulsed laser excitation at 480 and 980 nm will be discussed.

2. Experimental procedure

2.1. Growth using the μ-PD method

The μ-PD method was modified for the growth of fluoride crystals [15]. The concept is similar as for oxide compounds. The growth chamber can be evacuated up to 10^{-5} Torr by combined rotary and diffusion pumps. The growth chamber is equipped with a CaF_2 window for visual observation of the solid/liquid interface using a charge coupled device (CCD) camera with monitor. High-purity graphite crucibles were used and were inductively heated using a radio-frequency generator.

Starting materials were prepared from a stoichiometric mixture of 4N, KF, YF_3, PrF_3 and YbF_3 powders (Stella Chemifa Co. Ltd.). They were thoroughly mixed and put into the crucible. The chamber was evacuated to 10^{-4} Torr and the crucible was heated to 600 °C for 1 h to remove oxygen impurities. During this baking procedure, the chamber is further evacuated up to 10^{-5} Torr. After the baking, the recipient was filled with high-purity CF_4 (6N) until ambient pressure and the crucible was heated up to the melting temperature of about 1030 °C. Crystal growth was carried out using an *a*-axis seed of undoped KYF crystal. The growth rate was 0.1–0.15 mm/min. Controlling power and pulling rate during the growth process constantly maintained the crystal diameter. A photograph of the μ-PD furnace and the scheme of a typical thermal setup are given in Fig. 1(a and b), respectively. Samples, were cut, to 2.5 mm in diameter and thickness of 1.36 mm and polished for optical experiments.

2.2. Phase characterization

To identify the obtained phase, powder X-ray diffraction analysis was carried out in the 2θ range from 10° to 70° using a RIGAKU diffractometer (RINT2000). The X-ray source was CuKα with accelerating voltage 40 kV, and tube current 40 mA. All X-ray experiments were carried out at room temperature (RT) under air ambient. The crystallinity of the crystals was measured by X-ray rocking curve (XRC) analysis using a RIGAKU advanced thin film X-ray system (ATX-E). $CuK\alpha_1$ radiation was used with a multilayer X-ray mirror. The XRC profiles were taken with a four-bounce Ge (2 2 0) channel cut monochromator. The beam divergence was 12″.

The chemical composition was analyzed by electron probe microanalysis (EPMA) using a JEOL JXA-8600L analyzer. The distribution of Pr and Yb in the single crystal was measured along the growth axis using an electron probe of 10 μm in diameter and a step of 250 μm.

2.3. Optical measurements

Optical absorption spectra were recorded at RT with a Lambda 900 UV–viS–NIR Perkin-Elmer spectrometer. Fluorescence spectra under selective pulsed laser excitation in the $^2F_{5/2}$ multiples of the Yb^{3+} ($4f^{12}$) ground state configuration were measured using an optical parametric oscillator (OPO) pumped by a Nd:YAG laser. The energy of the beam impinging on the sample was always maintained around 1 mJ/pulse throughout the measurements. The excitation laser beam was focused onto the samples under glancing incidence in all cases to prevent the predominant part of the reflected laser light from being collected together with the fluorescent emission. Detection of the luminescence in the visible range observed at the geometry of 45° glancing angle was analyzed by a f-125 monochromator with a grating of 400 and 1200 grooves/mm

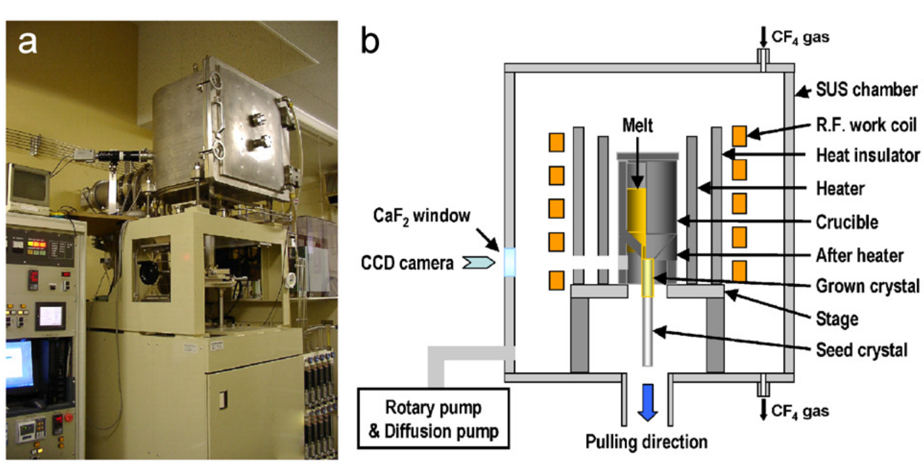

Fig. 1. Photograph (a) and schematic (b) of the μ-PD apparatus for fluoride crystal growth.

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