



Crystal growth, upconversion, and infrared emission properties of Er^{3+} -doped KPb_2Br_5

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Abstract

We report on the material preparation and optical properties of Er^{3+} -doped KPb_2Br_5 (KPB). KPB has a maximum phonon energy of only 138 cm^{-1} and is non-hygroscopic, which makes KPB an attractive candidate for solid-state laser applications. The preparation of Er doped KPB was based on a careful purification of starting materials followed by self-seeded Bridgman crystal growth. Under 975 nm diode laser pumping, Er:KPB revealed intense blue upconversion emission. For comparison, Er-doped KPb_2Cl_5 (KPC), which has a maximum phonon energy of 203 cm^{-1} , exhibited a dominant green Er^{3+} upconversion emission. The blue upconversion from Er:KPB can be attributed to emission from the $^4\text{F}_{7/2}$ excited state of Er^{3+} , which is quenched in most solid hosts due to strong multiphonon non-radiative decay. Due to the small phonon energy of KPB, the $^4\text{F}_{7/2}$ level becomes highly radiative with a room-temperature lifetime of $\sim 85\text{ }\mu\text{s}$ and an estimated quantum efficiency of $\sim 94\%$. For comparison, the $^4\text{F}_{7/2}$ decay time in Er:KPC was only $\sim 11\text{ }\mu\text{s}$ at room temperature and the radiative quantum efficiency was estimated to be $\sim 9\%$. Infrared (IR) emission bands were observed at $1.5\text{ }\mu\text{m}$ ($^4\text{I}_{13/2} \rightarrow ^4\text{I}_{15/2}$), $1.7\text{ }\mu\text{m}$ ($^4\text{I}_{9/2} \rightarrow ^4\text{I}_{13/2}$), $2.0\text{ }\mu\text{m}$ ($^4\text{F}_{9/2} \rightarrow ^4\text{I}_{13/2}$), $2.7\text{ }\mu\text{m}$ ($^4\text{I}_{11/2} \rightarrow ^4\text{I}_{13/2}$), $3.6\text{ }\mu\text{m}$ ($^4\text{F}_{9/2} \rightarrow ^4\text{I}_{9/2}$), and $4.5\text{ }\mu\text{m}$ ($^4\text{I}_{9/2} \rightarrow ^4\text{I}_{11/2}$), indicating the potential of Er:KPB for IR laser applications. The absorption and IR emission properties of Er:KPB were investigated in terms of transition linestrengths, branching ratios, radiative decay rates, and emission cross-sections using the Judd–Ofelt method.

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1. Introduction

The development of rare-earth (RE)-doped low-energy phonon host materials is of significant current interest for solid-state laser applications including direct ultraviolet and visible lasers, upconversion

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lasers, and mid-infrared (MIR) lasers [1–5]. Compared to traditional laser hosts based on oxides and fluoride crystals, RE activated low-energy phonon materials exhibit significantly reduced non-radiative decay rates between closely spaced RE energy levels [1–5]. The reduced multiphonon relaxation rates lead to higher emission efficiencies for known RE transitions as well as the possibility of RE emission bands at new wavelengths. Laser materials with intrinsic low maximum phonon energies include halides, sulfides, and chalcogenides.

Recently, potassium lead chloride (KPb_2Cl_5 , KPC) has emerged as a new low-energy phonon laser host for RE ions [4–17]. KPC is non-hygroscopic and readily incorporates trivalent RE ions. The maximum phonon energy of KPC is 203 cm^{-1} [4,9], which results in efficient MIR emission due to small multiphonon relaxation rates. Laser action has been demonstrated from Dy:KPC at $2.43\text{ }\mu\text{m}$ [7], Nd:KPC at $1.06\text{ }\mu\text{m}$ [8], and Er:KPC at 1.7 and $4.5\text{ }\mu\text{m}$ [15]. The overall efficiencies of the reported RE:KPC lasers, however, was relatively low ($<10\%$) mainly due to poor crystal quality and not optimized RE concentrations and pumping schemes.

We are currently investigating KPb_2Br_5 (KPB) as a potential new solid-state laser host material. Initial results of Nd-, Er-, and Tb-doped KPB were recently presented at conferences by several research groups [18–20]. KPB has a maximum phonon energy of only 138 cm^{-1} [18,19], which promises small non-radiative decay rates and high emission efficiencies for RE transitions with small energy gaps. In the following, we present results on the material preparation and optical spectroscopy of Er doped KPB including transmission and absorption, upconversion emission, infrared emission, and lifetime measurements. Using Judd–Ofelt (JO) theory, transition linestrengths, radiative lifetimes, branching ratios, and cross-sections were calculated and will be discussed.

2. Material preparation and basic material properties

The preparation of KPb_2Br_5 was based on commercial starting materials of PbBr_2 and KBr

with 99.999% purity. The PbBr_2 material was carefully purified through a combination of directional freezing and horizontal zone-refinement [21,22]. Subsequently, KPB was synthesized using stoichiometric amounts of purified PbBr_2 and KBr. The synthesized KPB material was further purified through zone-refinement. 1 wt% of ErBr_3 (99.9% purity) was then mixed with the purified KPB. The resulting material was loaded into a quartz ampoule and sealed under vacuum. The Er:KPB crystal was grown using Bridgman technique at a translation speed of $\sim 1\text{--}2\text{ mm/h}$. After the growth process, the resulting crystal was slowly cooled over a time period of 2 days. The Er concentration was measured to be $\sim 0.14\text{ wt}\%$ at Galbraith Laboratories using inductively coupled plasma optical emission spectroscopy (ICP–OES). For comparative spectroscopic studies, Er-doped KPC with 1 wt% ErCl_3 was prepared using a similar procedure as described before.

There are only a few reports in the literature on the crystal structure of KPB [23–25]. KPB belongs to a family of congruently melting ternary alkali lead bromides including KPb_2Br_5 , RbPb_2Br_5 , and CsPbBr_3 . According to Beck et al. [24], KPB is monoclinic (space group $\text{P}2_1/\text{c}$) with an angle β very close to 90° . The unit cell parameters are $a = 0.9264\text{ nm}$, $b = 0.8380\text{ nm}$, $c = 1.3063\text{ nm}$, $\beta = 90.06^\circ$, and $Z = 4$ [24]. The melting point of KPB is $\sim 382^\circ\text{C}$ and a phase transition occurs at 242°C [23]. The density of KPB is 5.53 g/cm^3 [25]. Pb^{2+} ions occupy two non-equivalent lattice sites of low symmetry, one site is a distorted octahedron and the second site a distorted trigonal prism [24]. Similar to RE-doped KPC, RE ions most likely occupy Pb^{2+} lattice sites in KPB with K^+ vacancies providing the necessary charge compensation [7–13].

3. Spectroscopic studies

3.1. Experimental details

The transmission measurements were performed using a Cary 5 spectrophotometer. Spectral emission studies were carried out using diode lasers operating at 660, 810, and 975 nm. The visible

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