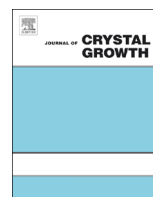




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# Micro-crystallization and spectroscopic properties of Er, Yb:RAl-borates ( $R=Y, Gd$ ) obtained in $Al_3(BO_3)_4-K_2Mo_3O_{10}-B_2O_3-R_2O_3$ and $Al_3(BO_3)_4-B_2O_3$ systems

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## ABSTRACT

The spontaneous  $YAl_3(BO_3)_4$  (YAB) and  $GdAl_3(BO_3)_4$  (GdAB) crystals (of nominal composition and co-doped with Er and Yb) up to 4–5 mm in length were grown from high-temperature solutions using  $K_2Mo_3O_{10}$  based fluxes in the temperature range of 1120–900 °C. Glass–ceramic composites based on the YAB and GdAB micro-crystals have been prepared by quenching  $YAB-B_2O_3$  and  $GdAB-B_2O_3$  melts and characterized by X-ray diffraction and spectroscopic methods. The vitrified melt was shown to contain micro- and nano-crystalline rare-earth borate phases. Their distribution over the composites has been investigated by electron microscopy and three-dimensional X-ray tomography. The absorption spectra of these materials co-doped with erbium and ytterbium as well as luminescence spectrum were demonstrated.

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## 1. Introduction

Lasers emitting at 1.5–1.6  $\mu\text{m}$  spectral range are of great interest for several industrial applications. This interest is first of all caused by the eye-safety of its radiation. Other advantage of this wavelength is high transparency in atmosphere and fused-silica waveguides. All this makes 1.5–1.6  $\mu\text{m}$  lasers very attractive for applications in eye-safe laser range-finders, LIBS systems, optical location and telecommunications.

The  $^4I_{13/2} \rightarrow ^4I_{15/2}$  transition of erbium ions is a simple and reliable method for obtaining 1.5–1.6  $\mu\text{m}$  laser operation. However, erbium ions suffer from low pump absorption at the wavelength of commercially available laser diodes near 980 nm. This fact obliges to use additional co-doping with ytterbium ions that strongly absorb pump radiation and transfer it to the erbium ions. For efficient operation of such Er–Yb co-doped lasers, two main spectroscopic conditions should be satisfied. The first is a short lifetime of the  $^4I_{11/2}$  energy level that prevents up-conversion processes and depopulation of this level by means of energy back transfer. The second condition is high enough  $^4I_{13/2}$  level lifetime to keep

quite low laser threshold. These conditions are well satisfied in Er, Yb:RAl-borates, which leads to the high interest in investigating the laser properties of these hosts [1–5]. However, the growth of these crystals with high optical quality and sufficient sizes ( $\sim 20 \times 10 \times 10 \text{ mm}^3$ ) for such applications remains a comparatively expensive process [6]. By this way the search for new relatively low-cost optical glass–ceramic composites with micro-crystals well-recommended before is promising [7–9].

## 2. Experimental

Complex fluxes based on potassium trimolybdate  $K_2Mo_3O_{10}$  with an addition of boron and rare earth oxides were used in all  $YAl_3(BO_3)_4$  (YAB) and  $GdAl_3(BO_3)_4$  (GdAB)-crystal growth experiments. Starting chemicals (at least 99.99% and 99.999% purity for rare earths and other materials, respectively) were  $Y_2O_3$ ,  $Gd_2O_3$ ,  $Yb_2O_3$ ,  $Er_2O_3$ ,  $Al_2O_3$  and  $B_2O_3$ , but  $K_2Mo_3O_{10}$  was previously sintered from  $K_2MoO_4$  and  $MoO_4$  according to reaction:



$K_2MoO_4$  was obtained by calcination of  $K_2MoO_4 \cdot 10H_2O$  in a Pt crucible at about 500 °C during 24 h, and  $MoO_3$  was prepared of  $H_2MoO_4$  on the same scheme.

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