Type of the Paper (Article)

Liquid-Phase Epitaxial Growth and Characterization of Nd:$YAl_3(BO_3)_4$ Optical Waveguides

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Abstract: We investigated fabrication of neodymium doped thin film optical waveguide-based devices as potential active sources for planar integrated optics. Liquid-phase epitaxial growth was used to fabricate neodymium-doped yttrium aluminum borate films on compatible lattice-matched un-doped yttrium aluminum borate substrates. We observed the refractive index contrast of the doped and un-doped crystal layers by differential interference contrast microscopy. In addition, characterization by X-ray powder diffraction, optical absorption and luminescence spectra demonstrated the crystal quality and uniformity and optical guiding of the resulting thin films.

Keywords: thin film crystal growth; epitaxial layer growth; multifunctional borate crystals; planar optical waveguides

1. Introduction

Integrated optics and photonics is increasingly important for optical signal processing in many applications, and relies on the development of compact, robust planar optical devices. Integrated optics employs waveguides as the building blocks for optical components which are then connected into circuits [1]. In particular, integrated optical systems using waveguide-based components typically include active devices such as lasers and modulators integrated into photonic circuits, and in each case, the structure must be designed to guide and confine the light within the active region of the device by careful design of the refractive index contrast between the cladding and the active layer. Optical waveguides are described as single mode or multimode based on the properties of the structure, as determined by the refractive index and dimensions of the guiding layer and substrate or cladding [2]. Active waveguide devices offer particular advantages over their bulk counterparts because the optical confinement increases the intensity of the signal and pump light within the waveguide, hence ensuring that the amplification or nonlinear optical frequency conversion can be more efficient than in comparable bulk devices [3]. Here we consider multimode active planar dielectric devices.

Fabrication of planar waveguide devices has been accomplished by a variety of approaches [3]. For example, the refractive index inside a dielectric material may be modified using nonlinear multiphoton processes [4, 5], or ion-exchange [6]. In another approach, two crystals may be optically polished and then thermally bonded to achieve strong adhesion between the crystal layers [7]. This typically requires careful polishing to ensure that the active layer is sufficiently thin. Epitaxial growth processes [8] such as liquid phase epitaxy (LPE) [9], pulsed laser deposition [10], molecular beam epitaxy (MBE) [11], hydrothermal epitaxy [12], metal oxide chemical vapor deposition (MOCVD) [13], or halide vapor phase epitaxy (HVPE) [14], enable production of high quality crystalline materials, which are practical for waveguide fabrication.

There has been long-standing interest in multifunctional doped borate laser crystals [10-19], for use in compact robust lasers emitting fundamental or self-frequency-doubled wavelengths, with Q-switched, mode-locked or continuous wave operation. The thermal conductivity of these crystals

facilitates the operation of the lasers at high power and in thin disk geometries [20-22], and un-doped borates have been adopted for nonlinear optics [23, 24]. This has led to a new drive for improved growth techniques for these crystals. Various approaches to optimize the crystal growth; the choice, preparation, and mixing of the flux, and the temperature profile, have been reported [10-12]. There is a balance between the relatively slow growth of the crystals, and the control of the crystal phase and uniformity, due to the formation of crystal twins [25, 26]. However, following an early report of epitaxial film growth [27], there has been recent interest to develop borate crystals for waveguide devices compatible with integrated optics, because this geometry enables concentration of the light in the active layer to enhance both the amplification and the optical nonlinearity of the device [28-31].

The liquid phase epitaxial growth technique has several advantages compared with other waveguide fabrication techniques. The layers are grown isothermally with homogeneous composition, so the quality of the epitaxial layer is comparable with that of bulk materials. The interface between the thin film and the substrate exhibits a step profile in the refractive index whereas other waveguide fabrication techniques typically lead to graded index profiles. Generally, the modes propagating inside a multimode step index profile structure have uniform effective index, whereas for a graded index profile, different modes have different effective index. The thickness of the waveguide structure can be controlled accurately by the growth duration and growth temperature. Finally, liquid phase epitaxy is adaptable for any single crystalline layer or active dopant, using an appropriate flux system and growth conditions [8].

We investigated liquid phase epitaxy as an effective growth method for Nd:$YAl_3(BO_3)_4$ (Nd:YAB) thin films on compatible lattice-matched un-doped borate substrates. The resulting films, whose lattice constants are consistent with R32 crystal symmetry, exhibit very good optical properties. Differential interference contrast microscopy, X-ray powder diffraction and optical absorption and luminescence spectra were used to characterize the optical quality and uniformity of these thin films.

2. Crystal Growth

2.1. Crystal growth methods

The flux system for the Nd:YAB epitaxial growth was chosen to be $K_2Mo_3O_{10}$ with excess Y_2O_3 and B_2O_3 [11]. This flux system offers advantages because it has lower volatility than the PbF_2-$3B_2O_3$ flux system, and excess Y_2O_3 and B_2O_3 were added to the initial flux to suppress Al_5BO_9 inclusions and to compensate for the volatility of B_2O_3 during crystal growth [11]. The mix for the growth was calculated as 8 at.% Nd/(Nd + Y) with 24.4 wt% of Nd:YAB in the solution. The solvent composition was 91.9wt% of $K_2Mo_3O_{10}$ + 5.4wt% B_2O_3 + 0.25 wt% Y_2O_3. All the chemicals were obtained from local suppliers, and heat treated in a 300 C furnace to remove adsorbed water before weighing. They were completely ground and mixed in the platinum crucible (5 cm diameter) and heated in an electric resistance furnace at 1150 °C for 24 hours. The temperature was then dropped to about °1000 C to find the actual saturation point by repeated seeding. The seed was settled to the mid part of the solution to ensure a homogeneous temperature gradient.

The substrate for the thin film growth needs to be selected carefully – it must permit reasonable lattice matching with the epitaxial layer to avoid strain due to lattice mismatch, and it must ensure a refractive index contrast, so that the active layer is the guiding layer with a higher refractive index. We selected un-doped $YAl_3(BO_3)_4$ (YAB) as it satisfies these criteria well. We also determined that a neodymium fraction of 8% permitted waveguide confinement, without excessive lattice mismatch. The calculated refractive index contrast for 8 at.% Nd dopant are 0.0632 for n_o and 0.0608 for n_e [11].

Figure 1 (a) shows the prismatic faces $\{11\bar{2}0\}$ and $\{2\bar{1}\bar{1}0\}$ and the rhombic face $\{01\bar{1}1\}$ for Nd:YAB, along with the crystal axes in Figure 1 (b). Previous liquid phase epitaxial growth of NdAB yielded thin films of good quality using $Gd_{0.59}La_{0.41}Al_3(BO_3)_4$ substrates with growth rates of around 1 µm/min [32].

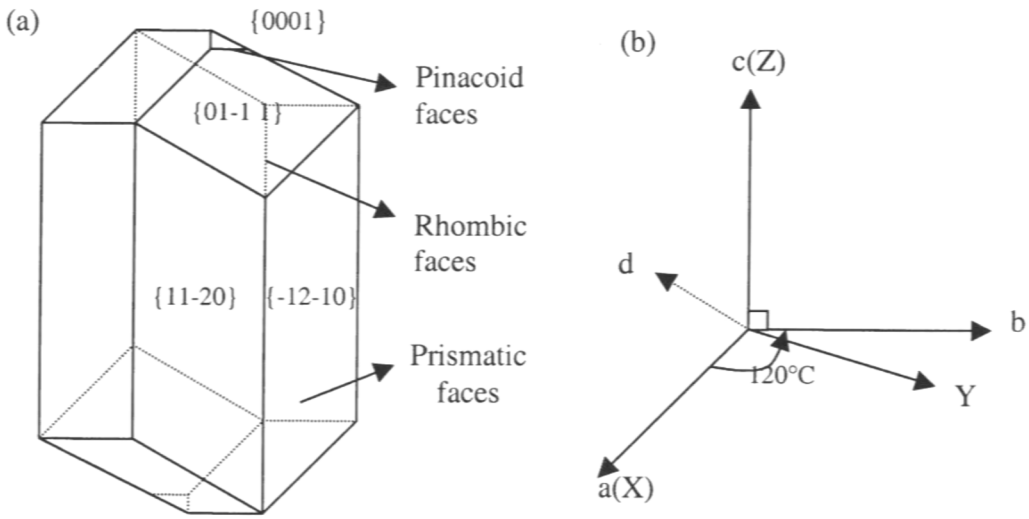

Figure 1(a) The growth habit of Nd:YAB crystals; (b) The hexagonal crystal axes for the crystals

The substrates were cut on the rhombic face $\{01\bar{1}1\}$ from bulk YAB crystals grown in our own laboratory and that of Prof. N. Leonyuk. The surfaces of the substrates were left unpolished. The substrates were dipped vertically into the melt with platinum wire wrapping the top of the substrates. The $\{01\bar{1}1\}$ cut pure YAB substrate (typical dimension 1 mm X 2 mm X 5 mm) was introduced and placed in the center of the flux in the crucible.

Liquid phase epitaxial growth of thin films is typically similar to that of bulk crystals. However the temperature is selected to be below the saturation point to allow thermodynamic growth of layers with the same orientation as the substrate while immersed in a super-saturated solution. At conditions which are close to equilibrium, the deposition of the crystal on the substrate is slow and uniform. In our case, the temperature was initially set to 1 °C above the saturation point to smooth and dissolve the surface (which becomes the substrate – film interface) and the temperature was then ramped down to 4 °C below the saturation point (around 1000 °C) to start thin film growth. The growth rate was about 5 µm per hour at this temperature, (see Figure 2). This growth condition was chosen to ensure the thermodynamic growth regime and avoid any risk of spontaneous nucleation (which appears to occur for ΔT of 8 °C or more). After growth, the substrate was carefully removed from the flux and slowly cooled down to room temperature over 24 hours. Bulk Nd:YAB crystals were also grown in our lab using the liquid phase epitaxy technique in the same solution. The solution mix was as for thin film growth, with a small YAB seed.

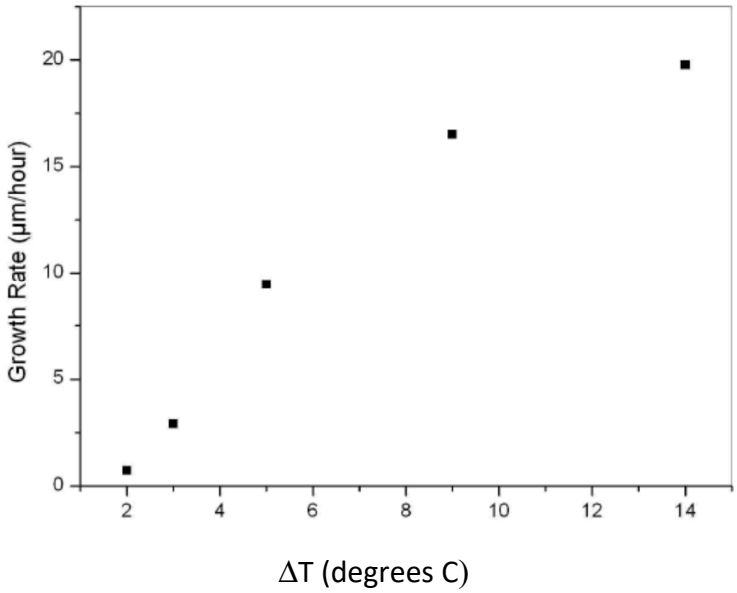

Figure 2: Nd:YAB crystal growth rate on rhombic face $\{01\bar{1}1\}$ seed crystal vs temperature below saturation.

2.2. Results of Crystal Growth

Figure 3a) shows an image of an as-grown Nd:YAB bulk crystal, with the growth facets visible, grown for two weeks under similar conditions as for the thin films, with the temperature ramping down by 0.5 C per day.. Figure 3b) shows an as-grown Nd:YAB crystalline thin film with un-doped YAB substrate that has been side-polished and imaged by differential interference contrast microscope (Olympus BX60). The Nd:YAB thin film layer appears as a uniform, smooth blue stripe in the microscope image with a sharp change and an apparent phase contrast with the pure YAB substrate, which suggests a step-like refractive index profile. The film thickness was measured to be 71 ± 0.5 μm. The film shown in Figure 3b) was obtained after epitaxial growth for 12 hours at the conditions specified above. The as-grown thin film sample was transparent and homogeneous with a smooth surface. No noticeable crystallites of the monoclinic form of Nd:YAB were obtained, as discussed in Ref [30]; we attribute this to the moderate Nd dopant fraction and lower temperature growth process that we used.

a) b)

Figure 3a). As-grown bulk Nd:YAB crystal, and Figure 3b) Differential interference contrast image of the Nd:YAB thin film and YAB substrate in cross-section.

3. Crystal Characterization Methods and Results

3.2. Crystal Characterization Results

X-ray powder diffraction was used to characterize the crystallographic structure and the results were compared with the diffraction patterns in an existing database. The X-ray powder diffraction (XRD) pattern of ground Nd:YAB bulk crystals grown by top-seeded solution growth was measured using a D/max-rA type X-ray diffractometer and CuKα radiation (λ = 1.54056 Å) at room temperature, and is shown in Figure 4. The X-ray powder diffraction pattern of the Nd:YAB crystals was found to be consistent with the reference pattern of $YAl_3(BO_3)_4$ (JCPDS card No. 15-117) [32], indicating that the neodymium dopant does not significantly perturb the lattice and the crystal belongs to the R32 space group. The lattice parameters were calculated by the least-squares method.

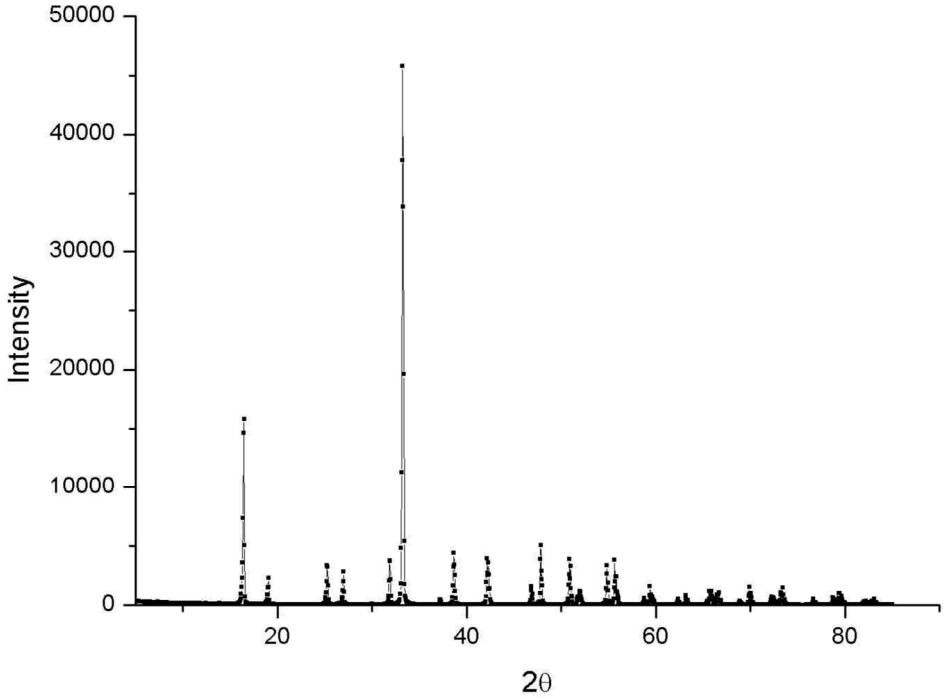

Figure 4: X-ray powder diffraction for as-grown Nd:YAB crystal

According to the X-ray diffraction data, the calculated lattice constants of the Nd:YAB crystals were $a = b$ = 9.298 Å, c = 7.2406 Å. In comparison with the data presented in Ref [11], the measured lattice constants of the bulk Nd:YAB crystal were close to those of $Nd_{0.09}Y_{0.91}Al_3(BO_3)_4$, which were 9.295 Å and 7.236 Å, which is consistent with 9 at.% N dopant concentration. Lattice constants of YAB crystals with different dopants are listed in Table 1. Assuming that the thin film has similar Nd concentration to that of the bulk crystal, the lattice mismatch of the as-grown Nd:YAB thin film is about 0.11% and 0.2% for the lattice constants a and c respectively. From optical microscopy and visual inspection the Nd dopant distribution in the film appeared uniform. This is expected as the solute concentration does not vary significantly during thin film growth.

Table 1. Lattice parameters of $YAl_3(BO_3)_4$

Crystals	*a* (Å)	*c* (Å)	*Reference*
YAB (JCPDS No. 15-117)	9.2872	7.2433	[32]
$Nd_{0.09}Y_{0.91}Al_3(BO_3)_4$	9.295	7.236	[11]
$NdAl_3(BO_3)_4$	9.365	7.262	[11]
Nd:YAB crystal	9.298	7.2406	This work

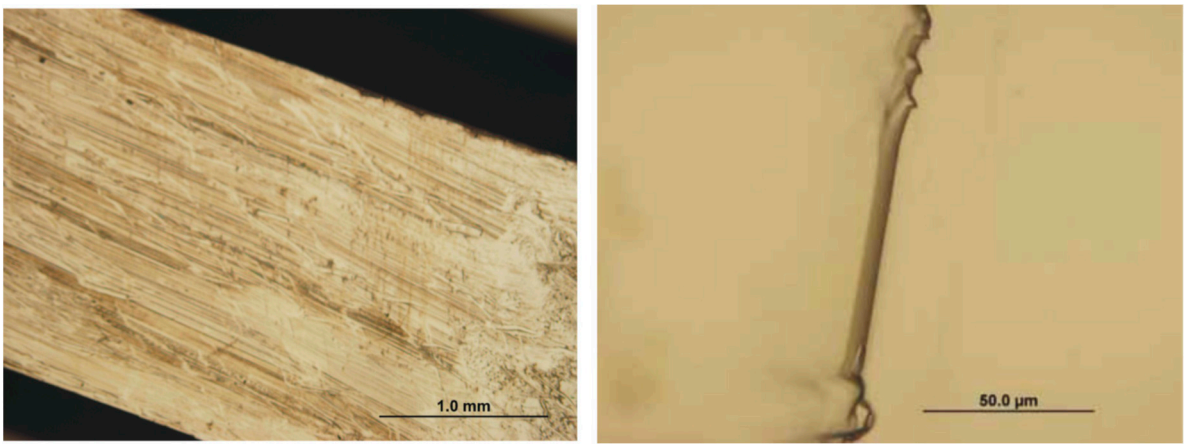

Figure 5 (a): Microscope images of substrate surface (5 ×) and (b) after growth thin film surface (20 ×) for Nd:YAB on a YAB substrate. The substrate is held vertically in the flux

The substrate crystal surface quality is shown in Figure 5a) before thin film growth, and it shows the rough unpolished surface, which was smoothed in the initial period of higher melt temperature. Figure 5b) shows the as-grown crystalline film image in cross-section. The epitaxial growth results in a smooth surface with no additional crystallite formation. For the purposes of crystal characterization, the absorption spectrum of a 2 mm × 4 mm × 1.1 mm slice cut and polished from a bulk Nd:YAB crystal was measured using a Cary 5E spectrophotometer. This was compared with the absorption along the guiding direction of the epitaxial layer as measured in the set up shown in Figure 6. Figure 7a) shows the (unpolarized) absorption spectrum of bulk Nd:YAB (thickness 1.1 mm) in the wavelength range 300 - 1000 nm. There are six main absorption peaks in the spectrum at 360, 528, 588, 750, 809, and 882 nm, which may be assigned according to Ref [11]. The uncorrected absorption spectrum for the thin film is illustrated in Figure 7b). The absorption peak positions and features are similar to those for the bulk sample. The drifting base line is attributed to the wavelength response of the detector in the Ocean Optics HR2000 spectrometer.

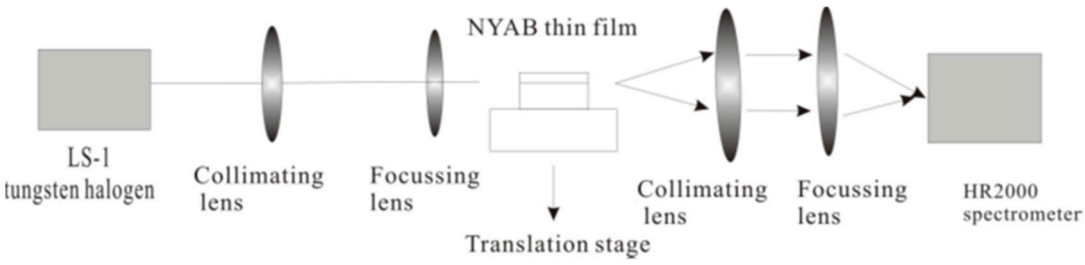

Figure 6: Experimental setup for measuring absorption spectra along guiding direction

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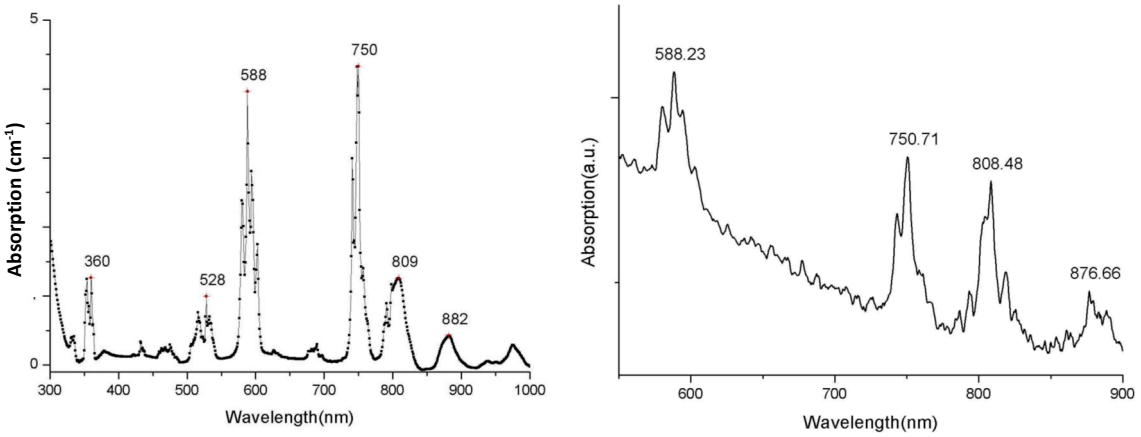

(a) (b)

Figure 7 (a) Absorption spectrum of a polished slice of bulk Nd:YAB and (b) Uncorrected absorption spectrum of Nd:YAB crystalline thin film, along the guiding direction, showing intensity peaks at 588, 750 and 808 nm, attributed to transitions from $^4I_{9/2}$ to $^4G_{5/2}$, $^4F_{7/2}$ and $^2H_{9/2}$ respectively.

The near-infrared fluorescence spectra of the Nd:YAB thin film (thickness 71 μm), and a bulk Nd:YAB crystal sample at room temperature are overlaid in Figure 7. This spectrum was obtained by coupling light from an 808 nm diode laser into the thin film and focussing the output by a lens into a fibre-coupled spectrometer. The 887 nm, 1062 nm and 1339 nm peaks correspond to the fluorescence of the $^4F_{3/2}$ level into the $^4I_{9/2}$, $^4I_{11/2}$, and $^4I_{13/2}$ multiplets, respectively. The room temperature fluorescence peak at 1062 nm is very strong, and the bandwidth (FWHM) of the 1062 nm peak is about 10 nm. The fluorescence spectrum of the thin film Nd:YAB crystal is well-correlated with that of the bulk Nd:YAB sample.

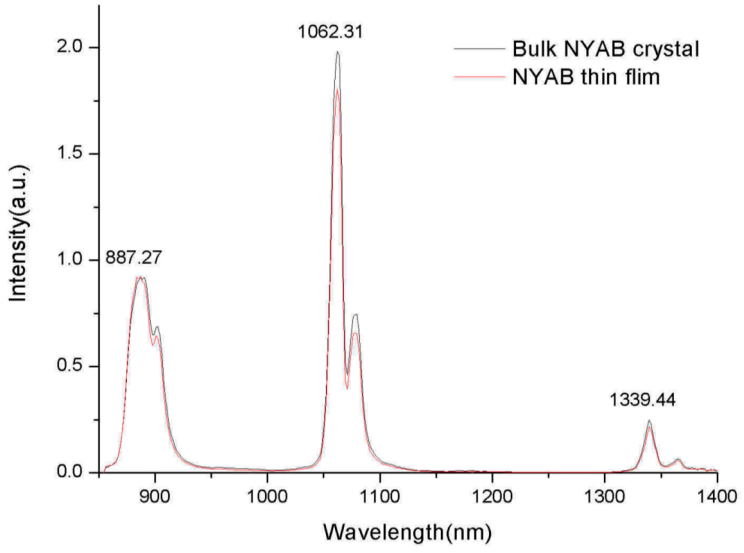

Figure 8: Luminescence spectrum of Nd:YAB thin film overlaid with that of the bulk Nd:YAB sample

A near-field image of the Nd:YAB thin film luminescence emitted from the end of the waveguide, as longitudinally pumped by the 808 nm diode laser, is shown in Figure 9. This was captured by a CCD camera (Pulnix) through a 1064 nm band pass filter. The camera was coupled to a laser beam analyser (Spiricon LBA100A). The luminescence image size is about 71 μm in the guided direction and 400 μm in the unguided direction. Two subsidiary images, probably due to back reflections, are also observable to the upper left. This image shows strong evidence of effective guiding within the epitaxial film layer.

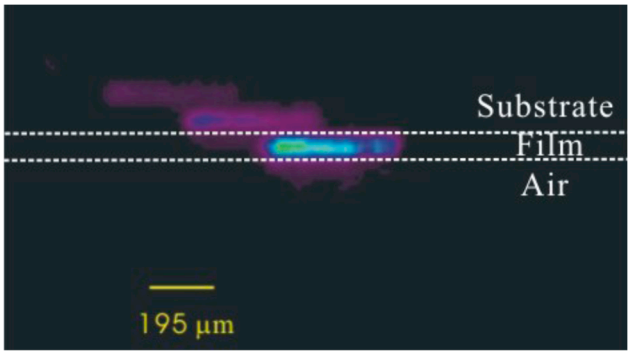

Figure 9 Luminescence image of Nd:YAB thin film, (image artefacts to the upper left).

4. Discussion and Conclusions

Nd:YAB planar waveguides with 9% Nd dopant were successfully grown by the top-seeded solution method from the $K_2Mo_3O_{10}$ and B_2O_3 flux system. The growth rates were measured and the growth conditions and procedure were selected for high quality film growth in the thermodynamic regime. Nd:YAB thin film layers were obtained at 4 °C below the saturation temperature with a growth rate about 5 µm/hour on an un-doped $\{01\bar{1}1\}$ YAB substrate. The growth of the thin films occurred over about 12 hours, with additional time for cooling. The as-grown thin films have good surface and optical quality and homogeneity, and exhibit effective waveguiding of the strong luminescence at 1062 nm. Future measurements of the optical gain and transmission losses in the devices are planned.

Author Contributions: Conceptualization JMD and YL; crystal growth methods and experiments YL; crystal characterization YL and PD; writing JMD and YL with editing by PD; project administration JMD.

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