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Overview of the laser and non-linear optical properties of calcium-gadolinium-oxo-borate $Ca_4GdO(BO_3)_3$

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Abstract

$Ca_4GdO(BO_3)_3$ (GdCOB) is a new non-linear optical (NLO) material which presents a congruent melting and can be grown from the melt in large size crystals (ϕ=50 mm, L=120 mm) using the Czochralski pulling method. This paper describes the crystal growth and NLO properties of GdCOB which compare favorably with those of commercial borates like BBO or LBO. Particular emphasis will be put on SHG of the Nd:YAG 1.064 μm laser emission. Large amounts of Nd or Yb ions can be substituted for Gd in this material and Ln:GdCOB with Ln=Nd, Yb exhibits interesting laser properties, especially in the case of diode pumped Yb activated crystals. Finally by combining the NLO properties of the GdCOB matrix and the laser emission associated with the active ion, a green self frequency-doubling laser is obtained. In this field, Nd:GdCOB appears the most promising material for practical applications, able to generate visible green laser light with only one single crystal instead of two, as usually. To date an Nd:GdCOB crystal yields 114 mW at 530.5 nm (for 1250 mW of absorbed pump power), when pumped with a 2 W high brightness laser diode. © 2000 Published by Elsevier Science S.A. All rights reserved.

Keywords: Non-linear-optical crystal; Self-frequency-doubling laser; Yb lasers; Diode-pumped laser; Green solid-state laser

1. Introduction

During the past 10 years, there has been an increasing interest in non-linear optical (NLO) crystals which allow the production of coherent light beams in a wide range of wavelengths. When pumped with laser radiations of frequencies ν_1 and ν_2, NLO materials may generate beams at $2\nu_1$ and $2\nu_2$ (second harmonic generation (SHG)), $\nu_1+\nu_2$ and $\nu_1-\nu_2$ (sum and difference frequency mixing (SFM, DFM)). Furthermore through optical parametric oscillation (OPO effect), it is possible with an NLO crystal to split an incident laser beam into two tunable beams (the 'signal' and 'idler' beams) whose photon energies sum is equal to the energy of the pump photon. In the near future, well-tuned solid state lasers such as Nd:$Y_3Al_5O_{12}$ (Nd:YAG) or diode pumped Nd:YVO_4 in conjunction with an NLO crystal, will probably be used instead of laser crystals doped with different lanthanide ions, to produce any required laser beam for a large variety of applications. This explains why the search for new NLO crystals has been very active in recent years. Several materials such as $KTiOPO_4$ (KTP), βBaB_2O_4 (BBO), LiB_3O_5 (LBO), $YAl_3(BO_3)_4$ (YAB) have clearly improved the NLO processes [1]. However, none of these crystals (except probably BBO) has a congruent melting. Consequently, they are grown using the flux method which is slow, produces crystals with limited size, usually rather expensive and sometimes of poor optical quality.

This has prompted us to search for new NLO materials exhibiting congruent melting which could be grown from the melt using the Czochralski pulling method. This technique is used to grow industrial crystals, such as the Nd doped YAG laser material. We mainly investigated borate compounds containing $(BO_3)^{3-}$ groups since the electronic delocalization in the planar borate anions was known to induce NLO properties (linked to their polar-

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izability) [2] as well as large birefringence (depending upon the relative orientation of the borate groups) required to fulfil the ‘phase matching’ conditions. One can also expect a broad transparency window, extending into the UV range [1]. Several points have guided our investigation. We were looking for a non-centrosymmetric compound (otherwise its second-order NLO susceptibility vanishes), non-cubic (birefringent) and which should exhibit congruent melting. Furthermore, since we were also interested in using such a material as a laser matrix for Nd^{3+} or Yb^{3+} ions, we add the requirement that its formula should contain rare earth ions such as La, Gd, Y, Lu, without any absorption band in the visible or infrared, that could be partially replaced by these fluorescent ions.

After several investigations, we came to the rare earth (RE) calcium oxo-borate family $Ca_4REO(BO_3)_3$ (RECOB) whose properties are reported in the following sections of this paper.

2. The RECOB NLO crystals

The $Ca_4REO(BO_3)_3$ phase exists for a number of RE^{3+} ions: La, Nd, Sm, Gd, Y, Er [3], Tb, Lu [4]. Their structure determined on tiny crystals grown from a PbO flux [3,4] is monoclinic (C_m space group). There are two kinds of isolated $(BO_3)^{3-}$ groups. They lie in planes perpendicular to [001] for one group and are skewed by ~30° for the other. There are also two types of distorted octahedral Ca^{2+} sites and RE^{3+} ions are located in the mirror plane of the monoclinic structure, in sixfold coordination with C_s site symmetry. Crystal growth experiments using melting zone and Czochralski methods were performed on the calcium rare earth oxoborates with RE = La, Y, Gd and Lu, the only ones without absorption lines in the visible and near IR spectral range. Single crystals were obtained with the first three ions [5] but GdCOB appeared as the easiest to grow. DTA experiments confirmed the congruent melting ($m_p = 1480°$). Consequently, GdCOB has been extensively studied in our group. Its crystal structure is given in Fig. 1. The initial results on crystal growth and NLO properties of GdCOB were presented in 1996 [6] and published later on [7,8]. Then other scientists undertook studies on YCOB: Sasaki et al. [9] grew YCOB crystals for NLO applications, while Chaï and co-workers [10,11] were primarily interested in the laser properties of Nd and Yb activated YCOB.

In Section 3, the present paper reviews the crystal growth and NLO properties of GdCOB. Section 4 will be devoted to the laser properties of Ln-doped GdCOB (Ln = Nd, Yb). Then, combining the laser and NLO properties of these materials, efficient self-frequency doubling laser emission in the green was achieved. These properties are described in Section 5 of this paper.

3. Crystal growth, linear and NLO properties of GdCOB

GdCOB powder was prepared by solid-state reaction at 1350°C from an intimate mixture of Gd_2O_3, $CaCO_3$ and H_3BO_3 or B_2O_3 in appropriate proportions. For the Czochralski crystal growth [8], ~250 g of GdCOB powder compressed into rods were melted in a 100-cm^3 iridium crucible under nitrogen atmosphere. Crystal growth started on a seed and proceeded at a growth rate of 0.5 to 1.5 mm h^{-1}. The crystal was rotated at 30–45 rev./min and approximately half the content of the crucible was converted into a GdCOB single crystal, typically 25 mm in diameter and 100 mm in length. Larger crystals (50 mm in diameter and 120 mm in length) were obtained at Crismatec using a larger iridium crucible (Fig. 2). Structural refinement confirmed the C_m space group and the very low average disorder on the large cation (Ca^{2+}, Gd^{3+}) sites [12]. The unit cell parameters that resulted from this refinement are $a = 8.078$ Å, $b = 15.98$ Å, $c = 3.55$ Å, $\beta = 101.28°$.

According to its monoclinic structure, GdCOB is a negative biaxial crystal. The orientation of the crystallophysic axes *X*, *Y*, *Z* with respect to the crystallographic directions was determined using X-ray Laüe and conoscopic patterns. Following the usual convention on the refractive index values $n_x < n_y < n_z$, the relative orientations are: $b // Y$; $(a, Z) = 26°$; $(c, X) = 15°$.

The dispersion curves of the refractive indexes were established on properly cut prisms using monochromatic sources and the minimum deviation technique. The results are given in Fig. 3, with the best fits obtained using single-pole Sellmeier equations [8]. These data allowed the calculation of the phase-matching curves for second harmonic generation, that is to say the directions, depending upon the incident laser frequency ν, at which the refractive indexes for frequencies ν and 2ν are identical (wave vector conservation law for SHG) [1]. The SHG process is cumulative only when this condition is fulfilled along the beam path in the NLO crystal. The results, in the three principal planes, are depicted in Fig. 4 for the two types of phase matching. In type I the two photons to be summed in the NLO crystal have parallel polarization and the resulting 2ν beam is polarized perpendicular to the ν one, while in type II the two photons at frequency ν have mutual perpendicular polarization and the beam at 2ν is polarized parallel to one of the ν photons.

These theoretical curves have been verified experimentally using a broad band femtosecond laser source [8]. It turns out that the SHG of the laser beam at 1.064 μm emitted by a Nd:YAG laser is possible only with type I phase matching and in the *XY* and *ZX* planes. A comparison of the SHG efficiency of GdCOB and BBO crystals around 1.2 μm, with BBO crystal taken as a reference, leads to an estimation of the effective non-linear

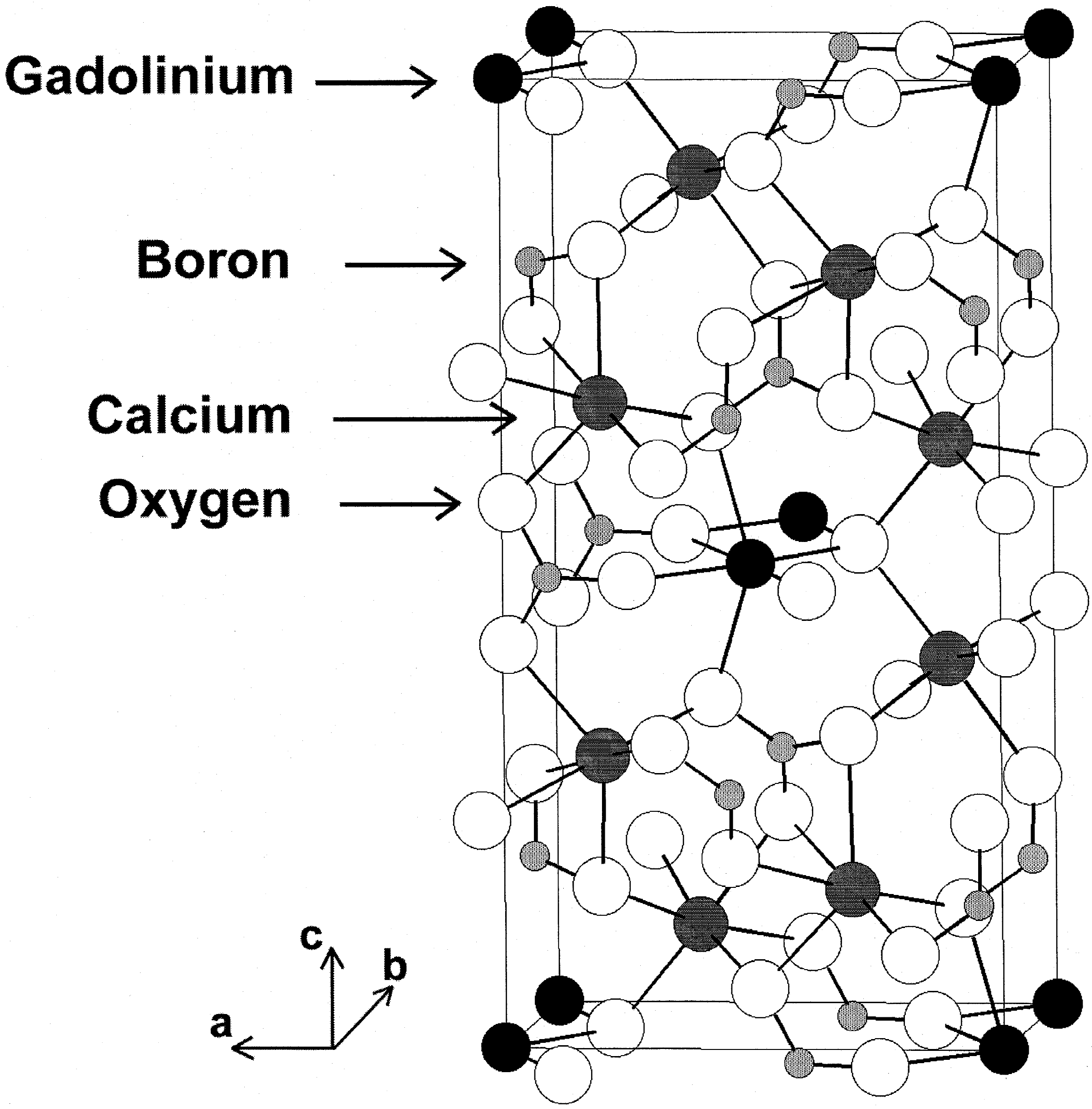

Fig. 1. Crystal structure of GdCOB.

coefficient d_{eff} in the phase matching directions. The d_{eff} values for GdCOB are: ~1 pm/V in the *ZX* plane and ~0.5 pm/V in the *XY* plane. The *ZX* d_{eff} value is equivalent to that of LBO and twice that of KDP (KH_2PO_4), the crystal commonly used for SHG in conventional Q switched flash-pumped Nd:YAG lasers. Tested with a commercial laser, a 15-mm-long GdCOB crystal has demonstrated SHG efficiency exceeding 50% [8]. Other parameters are of particular importance to characterize the interest of a NLO material used for SHG of the Nd:YAG laser. Some of them (angular acceptance, walk off angle) are related to the NLO process itself. Others, such as damage threshold, thermal conductivity, resistance to moisture . . . rule the possible development of a given material for practical applications. Table 1 summarizes the properties of GdCOB for SHG at 1.064 μm [7,8,12,13] compared to those of BBO and LBO [1,12]. With respect to the other borates, GdCOB can be grown in larger sized crystals because of its congruent melting and is expected to be cheaper when commercially available on a large scale. It presents a higher angular acceptance, a lower walk off angle and is non-hygroscopic. This is an excellent candidate to replace KDP and LBO for SHG of Nd:YAG lasers but it may be also promising for other NLO applications.

4. Laser properties of Ln:GdCOB, Ln = Nd, Yb

A large proportion of Gd^{3+} ions in GdCOB can be replaced by either Nd^{3+} ($4f^3$) or Yb^{3+} ($4f^{13}$) ions. Nd^{3+} is

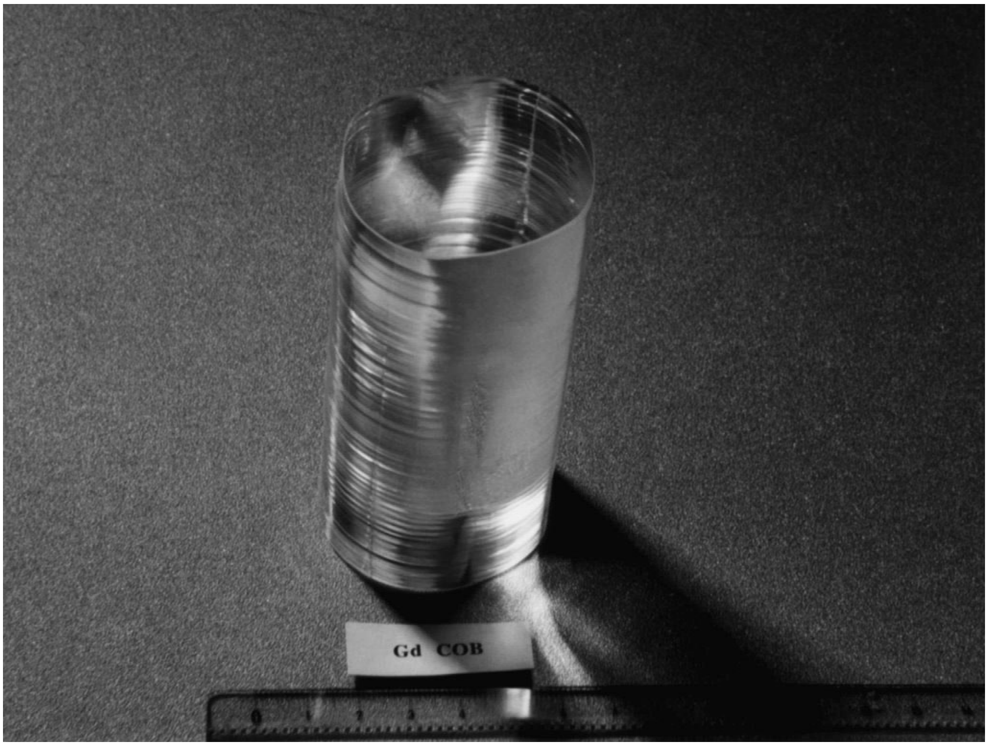

Fig. 2. A GdCOB single crystal grown from the melt by the Czochralski pulling method.

the most widely used activator ion in solid-state lasers [14] while Yb^{3+} appears now to be the actual Nd^{3+} challenger [14,15] for such applications. Both ions have an IR laser emission around 1 μm.

4.1. Nd:GdCOB

Several crystals containing up to 10% of Nd^{3+} ions (nominal) with respect to the total RE content were grown using the same process as for pure GdCOB. Then, cubes of these crystals with their edges parallel to the *X*, *Y* and *Z* indicatrix axes were cut and polished. For example, Fig. 5 gives the absorption spectrum of Nd^{3+} in GdCOB around 800 nm [16], the transition used to pump the Nd:GdCOB laser. One can notice the strong influence of the incident beam polarization. However, for all of them, the absorption cross section peaks at 811 nm ($\sigma_{abs} \sim 2\times10^{-20}$ cm^2). The Nd^{3+} fluorescence lifetime remains constant and equal to 98 μs for low doping rates (up to 9×10^{19} Nd^{3+} cm^{-3}). Then it decreases indicating that cross relaxation processes occur in this matrix. A Judd–Ofelt analysis of the absorption line strengths [16] allows to predict an Nd^{3+} radiative lifetime of ~660 μs. It means a quantum efficiency of the $^4F_{3/2}$ fluorescence $\eta = 98/660 = 15\%$. The multiphonon relaxation efficiency, related to the high phonon cut-off energy (stretching vibrations of the $(BO_3)^{3-}$ groups; $h\omega \sim$ 1250 cm^{-1}) in GdCOB, explains this rather low η value. The maximum emission ($^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition) of Nd:GdCOB occurs at 1.061 μm with emission cross section lying between 2 and 4×10^{-20} cm^2 according to the polarization and the direction of propagation.

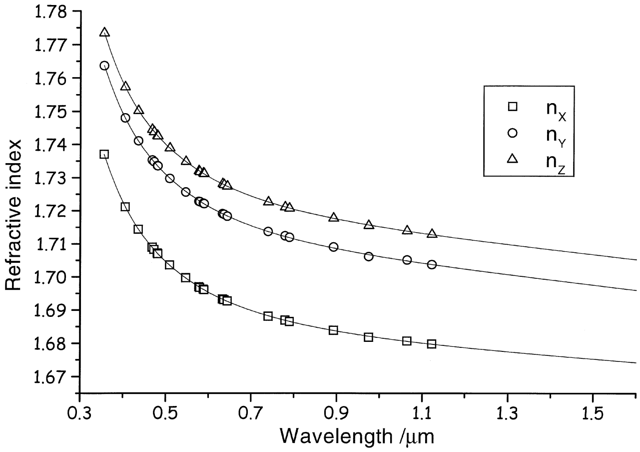

Fig. 3. Refractive index dispersion curves. The experimental data (points) were fitted using single pole Sellmeier equations.

Under titanium–sapphire laser excitation, laser action was obtained for pumping along the three principal directions [7,16]. The *Y* direction gives the best results (slope efficiency 34%, laser threshold 90 mW) for a 7% Nd^{3+} doped crystal. Decreasing the Nd^{3+} content to 4% (1.8×10^{20} ions cm^{-3}) improves the slope efficiency which

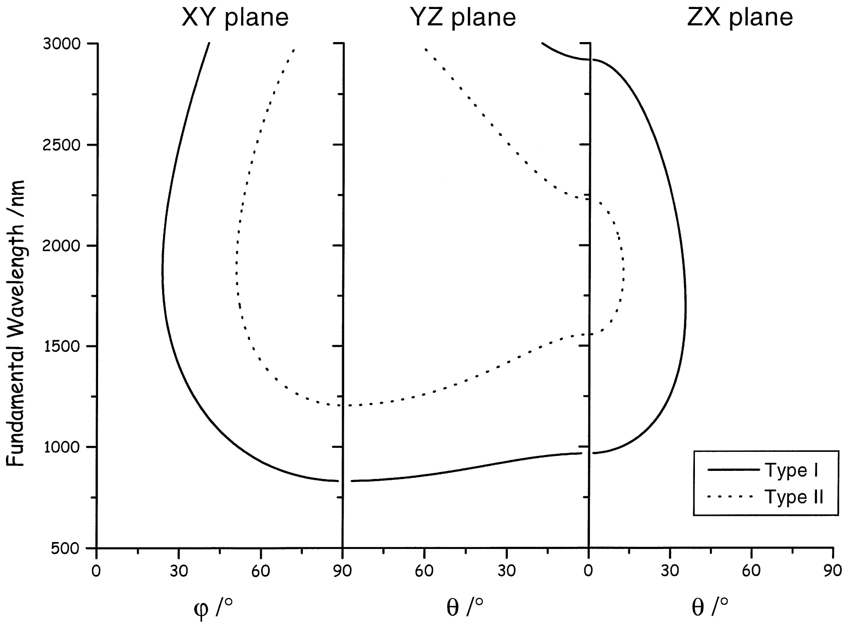

Fig. 4. Phase-matching curves for SHG in the three principal planes. Solid curve, type I; dashed curves, type II.

reaches 45% at the expense of a higher laser threshold [16]. In this latter case, about 400 mW of 1.061 μm emission were obtained for about 1100 mW of absorbed pump power. For sake of comparison, a Nd:YAG crystal pumped in the same cavity gave a slope efficiency of 46.5% but the threshold was only ~15 mW. Of course Nd:GdCOB would not compete with Nd:YAG as far as laser emission at ~1.06 μm is concerned, since its thermal conductivity is about five times smaller. However, the laser slope efficiencies of the two materials at low power emission are almost identical. This led us to combine the laser emission of Nd^{3+} ions and the NLO properties of GdCOB in order to generate directly a green laser beam with Nd:GdCOB through internal SHG of the infrared Nd^{3+} laser emission. It will be shown in Section 5 that this phenomenon does occur and is very efficient.

Table 1
Comparison between BBO, LBO and GdCOB properties. The NLO characteristics are given for type I SHG at 1.064 μm

	BBO	LBO *X* direction	GdCOB *ZX* plane
d_{eff} (pm/V)	2	1	1
Walk off angle (mrd)	70	7	13
Angular acceptance (mrd cm)	1.4	1.3	2.6
Resistance to moisture	Poor	Fair	Excellent
Crystal growth	Flux	Flux	Czochralski
Damage threshold 532 nm, 6 ns, 10 Hz (GW cm^{-2})	~1	~1	~1
Band gap transmission range (μm)	0.2–2.6	0.16–2.6	0.21–2.7[a]
Thermal conductivity (W m^{-1} K^{-1})	1.6	3.5	2.2

[a] For GdCOB, Gd^{3+} absorption lines occur around 250, 275 and 310 nm.

4.2. Yb:GdCOB

The Yb^{3+} ion has a very simple electronic structure with only two energy levels: $^2F_{7/2}$ (ground state) and $^2F_{5/2}$. Therefore Yb^{3+} doped crystals operate according to the pseudo-three level laser scheme [17]. The absorption and fluorescence spectra of Yb:GdCOB crystals are given in Fig. 6. These spectra extend over a broad spectral range, as a result of the combination of high crystal field splitting of the energy levels (1003 cm^{-1} for $^2F_{7/2}$), distribution of Yb^{3+} ions on Gd^{3+} and Ca^{2+} sites, and extensive vibronic coupling with the lattice phonons [15]. The 'zero line' lies at 976 nm and the Yb^{3+} fluorescence lifetime is 2.6 ms.

The best laser results were obtained using a 3-mm-long 15% doped Yb:GdCOB crystal. Under titanium:sapphire pumping at 902 nm, laser action was achieved at 1056 nm in this crystal with a 59% slope efficiency and a 40 mW laser threshold [15]. However, the most interesting results come from the fiber coupled InGaAs laser diode pumping experiments [18]; 191 mW of laser power at 1050 nm were obtained for 815 mW of incident power (slope efficiency: 42%). Moreover, the laser output decreases by only 30% when the temperature of the Yb:GdCOB crystal is increased by 50°C and the emission can be tuned from 1035 to 1088 nm using a Lyot filter. When replacing the output coupler by a low transmission mirror, the tunability of the laser was increased to 102 nm (1013 to 1115 nm) [18].

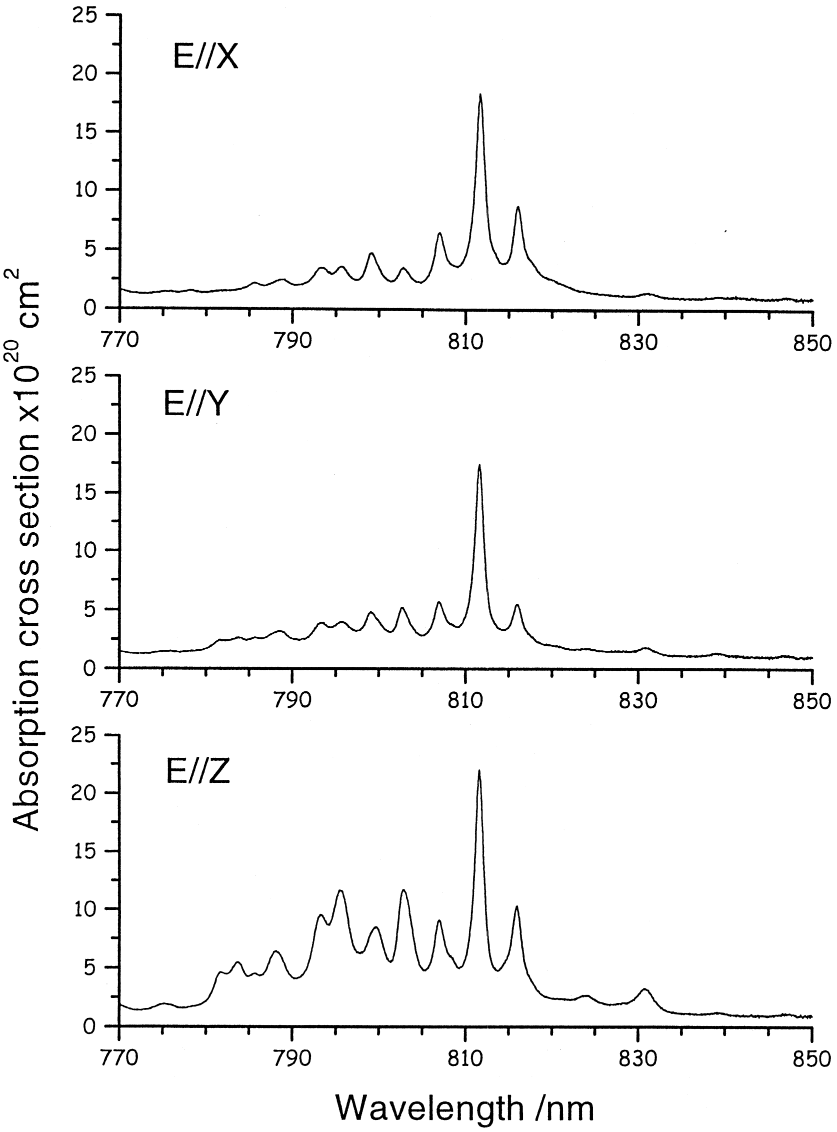

Fig. 5. Polarized absorption spectra of Nd:COB at 300 K; $^4I_{9/2} \rightarrow {}^4F_{5/2}, {}^2H_{9/2}$ transition around 800 nm.

These results reflect the large splitting of the Yb^{3+} $^2F_{7/2}$ ground state, demonstrating the interest of this new Yb activated laser material.

5. Self frequency doubling laser experiments

Thanks to the NLO properties of the GdCOB matrix, self frequency doubling laser emission was expected in Ln:GdCOB, Ln=Nd, Yb. Internal frequency doubling of the Nd^{3+} laser emission in the non-linear host lattice was indeed observed at the earliest stages of investigation of the laser properties of Nd:GdCOB [7]. As already mentioned, GdCOB SHG of a 1.06 μm laser beam is possible in the *XY* and *ZX* planes. However, during the investigation of the Nd:GdCOB laser emission at 1.061 μm, the laser beam polarization was found along *Z* or *X*, but never along *Y*. This prompted us to cut a 7% Nd:GdCOB crystal for SHG in the *XY* plane, even if it was not the most favorable plane regarding the value of the non-linear optical coefficient.

Such a crystal (AR coated for 1061 and 530.5 nm, length: 8 mm) gave at first 17 mW of 530.5 nm green laser emission [19] when pumped at 811 nm with a titanium–sapphire laser. By improving the laser cavity set up, the green output power (Fig. 7) was raised to 64 mW for 940 mW of absorbed pump power [20]. It can be seen in Fig. 7 that the green beam power goes quadratically with the absorbed pump power as expected for a SHG process. Furthermore under pumping with a 2 W high brightness laser diode, 114 mW of green self frequency doubled laser light was obtained for 1250 mW of absorbed pump power [20,21]. Up to now, these outstanding results are only exceeded by the best self frequency doubling laser crystal

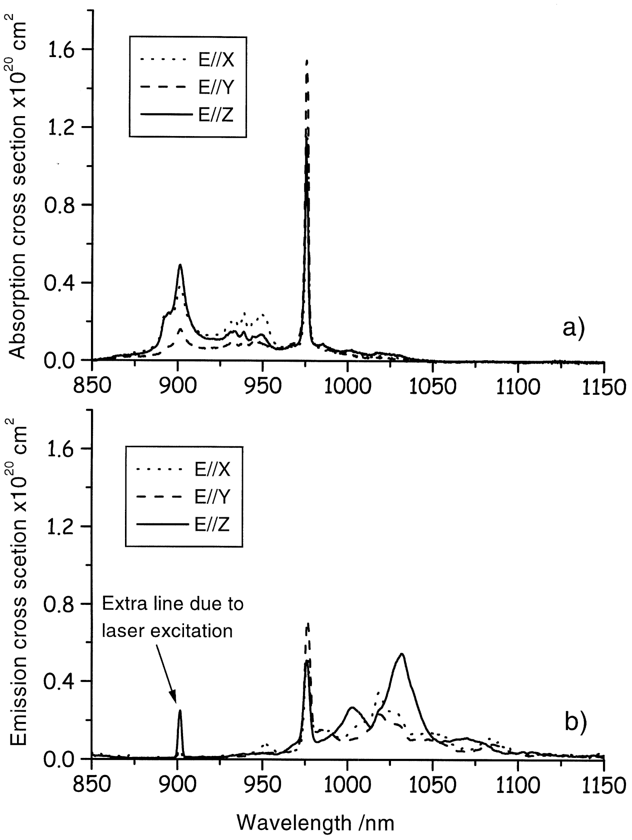

Fig. 6. Polarized absorption (a) and emission (b) spectra of Yb:GdCOB at room temperature.

Nd:YAB (NYAB) [22], which gives 160 mW of green light under similar diode pumping conditions. However NYAB is a non-congruent melting compound. Therefore, it has little future for practical applications where manufactured crystals are needed. Self frequency doubling laser emission for Yb:GdCOB was also observed [15]. Crystals containing 7% and 15% Yb^{3+} ions with respect to the total RE content were cut for SHG at 1043 nm in the *XY* plane and tested under titanium sapphire pumping at 902 nm. Green laser emission was actually observed, but it was very unstable owing to the broad tunability range of the Yb:GdCOB laser. Indeed, to decrease the losses due to frequency conversion, the laser shifts its IR emission to another wavelength for which the phase matching conditions are no longer fulfilled for the studied crystal. Further experiments using wavelength selective elements in the laser cavity set up are in progress.

6. Conclusion – prospective

GdCOB and Ln:GdCOB single crystals appear very efficient materials for non-linear optical and laser applications. GdCOB presents several advantages with respect to KDP or other commercial borate crystals such as LBO or BBO and could replace them for SHG of the Nd:YAG laser or for other NLO applications. For instance the use of GdCOB crystals for OPO should be considered. Nd:GdCOB crystals operating as self frequency doubling lasers are already very attractive solid state green coherent sources. With only one single crystal, they give rise to laser performance at low power similar to those obtained usually with two crystals, one for laser action and the other for SHG.

Yb:GdCOB crystals are also interesting laser materials because of their large slope efficiency under diode pumping and broad tunability range. These latter properties could allow matching a specific wavelength, for instance for helium spin polarization (1083 nm), or to generate ultra-short laser pulses.

Other members of the RECOB family are of interest. As an NLO material YCOB, although slightly more difficult

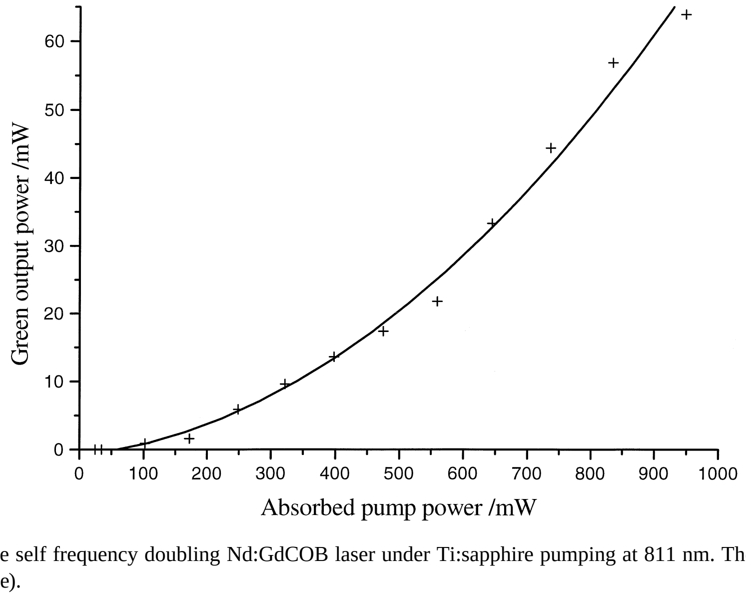

Fig. 7. Output–input curves for the self frequency doubling Nd:GdCOB laser under Ti:sapphire pumping at 811 nm. The experimental points (crosses) are fitted to a quadratic law (solid line).

to grow than GdCOB, has some specific interests [9,13,23]. It is slightly more birefringent and for instance can give type I and type II SHG of the 1064 nm Nd:YAG laser emission. This material can also be used to produce the third harmonics of the Nd:YAG laser beam (355 nm) through sum-frequency mixing (1064 nm + 532 nm).

The investigation of the NLO and laser properties of the RECOB family is expanding and new results are expected in the near future in the different groups concerned with the study of these compounds.

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