Contributed paper

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Oxide crystals for solid state laser applications

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Recent trends in optoelectronics are towards small solid state lasers pumped by laser diodes. The paper describes efforts to create material basis for the development of this branch of optoelectronics. Single crystal growth and properties of many compounds acting as active laser or self-frequency doubling materials, passive modulators, nonlinear optical materials, and acousto-optic materials are discussed, including yttrium aluminium garnet with high neodymium concentration, vanadates, borates, and double tungstates.

Keywords: oxide single crystals growth, diode pumped solid state lasers, nonlinear absorbers, laser frequency conversion, acousto-optics, Nd:YAG, CLBO, BBO, GdCOB, Nd:YVO, Nd:KGW, substrates for GaN epitaxy, TeO_2.

1. Introduction

Development of laser techniques and practical applications of lasers in many areas of materials research and technology requires a great variety of materials possessing suitable properties. The intensive search for new crystals with good optical quality, very high structural perfection, and improved lasing properties for applications both in bulk and microlasers is still carried on. The paper describes investigations of crystallisation of oxide materials as well as their characterisation as potential candidates for solid state laser applications carried out in Institute of Electronic Materials Technology (IEMT), Institute of Applied Physics (IAP), and Institute of Optoelectronics (IOE). The three institutes have been collaborating for last decade, making efforts to create the material basis for development of optoelectronics in Poland. As a result of this co-operation technologies of many oxide single crystals such as garnets, vanadates, borates, and tungstates, among others, have been developed.

IEMT investigates crystallisation of congruently melting oxide compounds with the use of the Czochralski technique. The furnaces with controllable atmospheres, r.f. heating, and diameter control systems allow to grow single crystals of materials melting at high temperatures (up to 2000°C). On the other hand IAP uses resistance heating for crystallisation of oxide compounds with melting points up to 1400°C. Owing to very good temperature stabilisation and possibility of shaping the temperature conditions in a very broad range, the resistance furnaces are used both to the Czochralski growth of congruently melting materials and for high temperature solution growth (HTSG) of incongruently melting compounds. The latter technique is also used for crystallisation of materials with high temperature irreversible phase transitions – the use of the flux allows to lower the temperature of crystallisation below the temperature of the phase transition. IOE carries out full characterisation of optical properties of as grown single crystals, what enables us to choose proper modifications of grown crystals by introducing proper amounts and types of dopants as well as to build optoelectronic devices.

In the following sections we described active laser materials, nonlinear optical materials for laser frequency conversion, materials for Q-switched passive modulators, and acousto-optic materials for laser light modulation.

2. Active laser materials

Oxide single crystals are widely used in solid state lasers as active materials. Owing to selective pumping by means of laser diodes, laser technique has been revolutionised in recent decade. Solid state lasers with diode pumping form a new class of lasers having favourable properties and wide applicability. The matching of laser diodes emission bands and absorption bands of active materials leads to high pumping efficiency, single-mode laser radiation (shear or longitudinal), and miniaturisation of lasers. Such lasers currently replace other types of lasers and find applications in such areas as interferometry, spectroscopy, laser metrology, range finding, and military devices. One can expect that due to continuous improvement of their properties, possibility of complex matrix formation, and diminishing prices diode pumping soon become the commonly used technique of solid lasers excitation.

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Researches of new materials having optimal macroscopic (good mechanical and thermal strength) and microscopic (structure allowing introduction of active ions) properties as well as of potential activators from lanthanides (Nd^{3+}, Er^{3+}, Yb^{3+}, Ho^{3+}, etc.), actinides (U^{2+}) and transient elements (Cr^{3+}, Cr^{4+}, Ti^{3+}, Co^{2+}) are still carried on. High level of doping allows increasing the pumping efficiency due to widening of absorption bands in the pumping area, so crystals that can be heavily doped are of great interest. On the other hand, heavy doping is often limited by concentration quenching of luminescence, which can decrease the lifetime of upper laser levels. Our investigations of active materials took these two factors into consideration to find a compromise solution between need of high pumping efficiency and properly long lifetime of upper levels. In the following sections several crystals are described. They are gathered in two groups; active laser crystals (YAG:Nd, YAG:Er,Yb, YVO_4:Nd, $KGd(WO_4)_2$:Nd – subsection 2.1) and the crystals having also nonlinear optical properties, what gives in result the possibility of concurrent laser action and frequency doubling in the same crystal (self-doubling frequency crystals, $Ca_4GdO(BO_3)_3Nd$ and $YAl_3(BO_3)_4$:Nd – subsection 2.2).

2.1. Active laser crystals

Nd:YAG single crystals

Neodymium doped yttrium aluminium garnet crystals $\{Y_3\}[Al_2](AlO_4)_3$, known as Nd:YAG, have become the most popular active laser material. They were discovered nearly 35 years ago [1]. Progress in crystallisation by the Czochralski technique, due to a growth process controlled by computer, has so improved quality of YAG crystals that Nd:YAG lasers have been applied in many different areas of optoelectronics.

Nevertheless YAG crystals seem not to be an ideal host for doping with Nd^{3+} ions [2]. Generally it is difficult to introduce above 1 at% of Nd homogeneously into the YAG lattice because the effective segregation coefficient is ~0.2. Neodymium dopant locates in dodecahedral sites instead of Y^{3+} ion sites and large ionic radius of Nd^{3+} is not favourable to this exchange. Usually amount of Nd^{3+} that incorporates into the YAG lattice is rather small. This mismatch is the reason of difficulties in forming a solid solution of $Nd_3Al_2(AlO_4)_3$ with $Y_3Al_2(AlO_4)_3$. In addition the distances between cation lattice positions for the aluminium garnets are small what enables ion-ion interaction producing, as a result, concentration quenching of the Nd^{3+} fluorescence.

Higher concentrations of Nd^{3+} ions were introduced (0.3–4.8% at Nd) into Nd:YAG ceramics but not into single crystals. Nd:YAG crystals obtained quite recently contain 1.1–1.76 at % of Nd^{3+} ions. Crystals were grown by the Czochralski method using Cyberstar apparatus. The growth process and crystal diameter were controlled at a programmed rate by means of weighing of the growing crystals. The crystals grew in the [111] direction. Typical core formation (3 mm in diameter) in the central part of crystal due to growth with convex interface was observed.

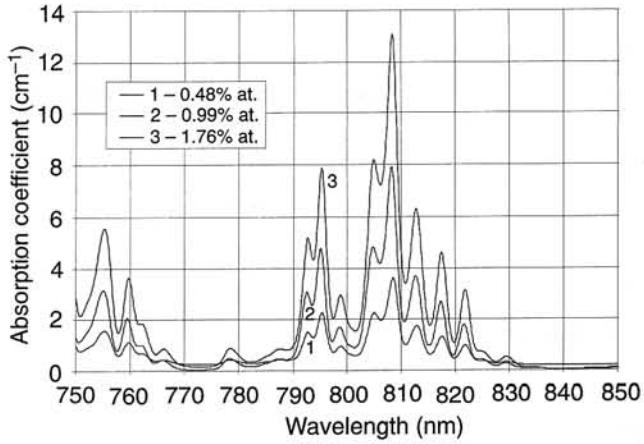

Fig. 1. Spectra of the absorption coefficient of neodymium doped YAG crystals (1–0.48 at.% Nd^{3+}, 2–0.99 at.% Nd^{3+}, 3–1.76 at.% Nd^{3+}).

Good quality of as-grown YAG:Nd single crystals without inclusions and bubbles was confirmed by elastoscopic method and Mach-Zehnder interferometer measurements. The absorption spectrum of the obtained YAG:Nd crystals was investigated in the range 200–1100 nm with the resolution $\Delta\lambda$ = 1 nm and in the range 750–850 nm ($\Delta\lambda$ = 0.1 nm) using the Perkins Elmer Lambda 2 Spectrophotometer. The absorption coefficient was calculated taking into account multiple reflections inside the sample. Figure 1 presents absorption spectra of YAG:Nd containing 0.48, 0.99, and 1.76 at % of Nd^{3+} ions, Fig. 2. – luminescence spectrum of the neodymium doped YAG crystal (1.76at%), and Fig. 3 shows fluorescence decay time versus concentration of neodymium in YAG.

Er:YAG, (Er,Yb):YAG crystals

In the last years, interest in developing of lasers emitting in range near 1.55 μm has been observed. The wavelengths of ≈1.55 μm are called eye-safe because high pulse energies can be used without danger of eye damage. This kind of emission can be used in telecommunication, range finders

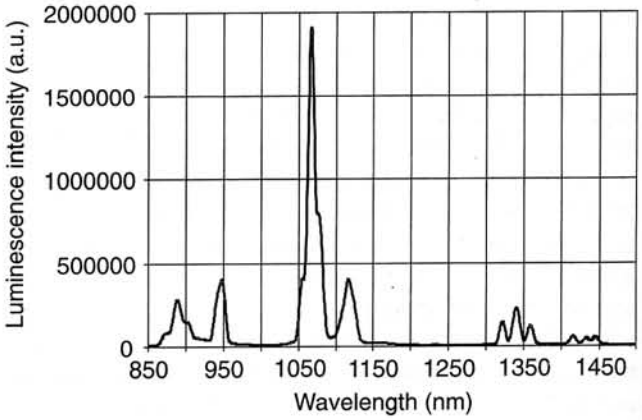

Fig. 2. Luminescence spectrum of the neodymium doped YAG crystal (1.76 at.% Nd^{3+}).

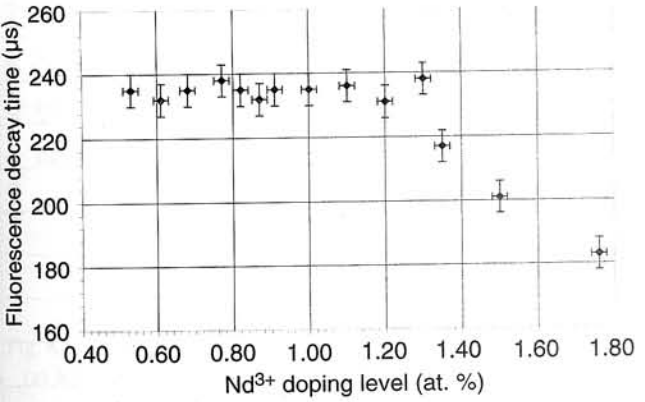

Fig. 3. Influence of doping level of Nd^{3+} ions in YAG:Nd^{3+} crystals on fluorescence decay time ($^4F_{3/2}$).

and other devices. Er^{3+} and Er^{3+}, Yb^{3+} doped YAG crystals are suitable for this application.

The single crystals were grown using the same technique and equipment as in the case of Nd:YAG. The crystals were grown in the [111] direction and typical for garnet crystals core formation was observed. Elastoscopic method and Mach-Zender interferometer measurements were used to examine the quality of crystals. Absorption measurements in range 200–1100 nm were done in the same way as for Nd:YAG crystals and absorption coefficient was calculated. Figure 4 shows absorption spectra of erbium and erbium-ytterbium doped YAG crystals and Fig. 5 shows luminescence spectra of these crystals [3,4]. The fluorescence decay times of the examined samples are listed in Table 1.

Table 1. Fluorescence decay times (level $^4I_{13/2}$) of the investigated YAG:Er^{3+} and YAG:Er^{3+},Yb^{3+} crystals.

Crystal	Fluorescence decay time
YAG:Er^{3+} (1.5% Er^{3+})	5.6 ±0.1 ms
YAG:Er^{3+},Yb^{3+} (0.9% Er^{3+}, 9% Yb^{3+})	4.3 ±0.1 ms
YAG:Er^{3+},Yb^{3+} (0.5% Er^{3+}, 5% Yb^{3+})	3.5 ±0.1 ms

Nd:YVO_4 crystals

Neodymium doped yttrium orthovanadate Nd:YVO_4 crystal is one of the most efficient laser hosts for diode pumped solid state lasers. Nd:YVO_4 lasers have the advantages of low dependence on pump wavelength and temperature control of diode laser, high slope efficiency, low lasing threshold, linearly polarised emission, tendency to the single mode output and large stimulated emission cross-section at both 1.06 μm and 1.3 μm.

Growth of yttrium orthovanadate crystals is connected with some difficulties. At the melting point at 1810°C stoichiometric YVO_4 loses oxygen very rapidly and composition of the melt continually changes. First, YVO crys-

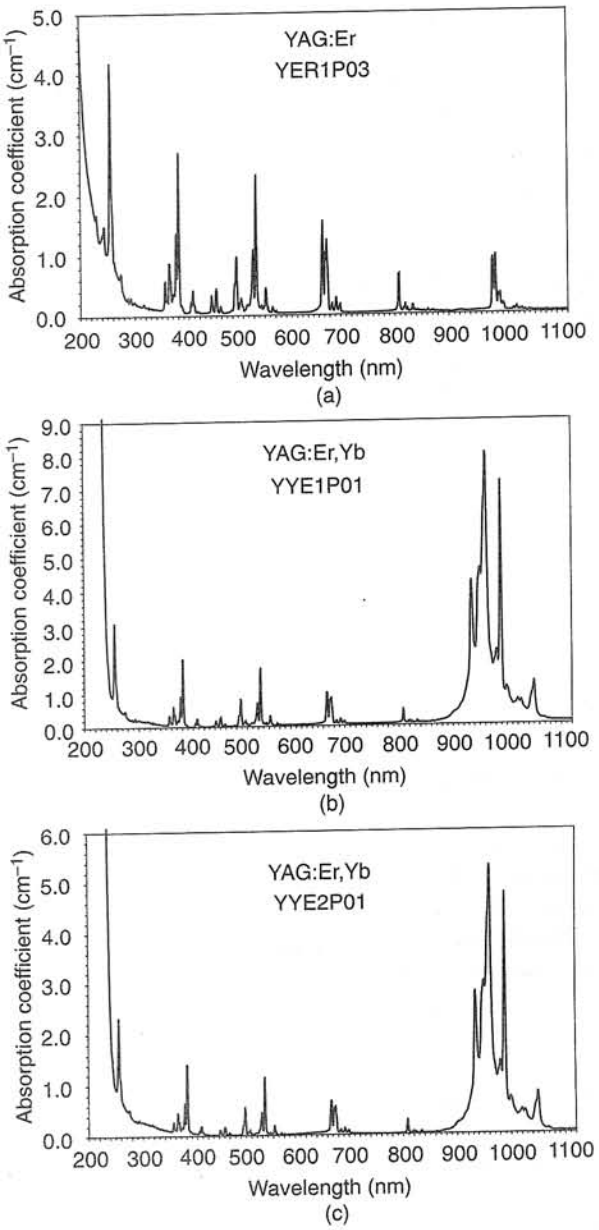

Fig. 4. Spectra of the absorption coefficient of erbium and ytterbium doped YAG crystals: (a) YAG:Er^{3+} (1.5 at.% Er^{3+}), (b) YAG:Er^{3+},Yb^{3+} (0.9 at.% Er^{3+}, 9 at.% Yb^{3+}), YAG:Er^{3+},Yb^{3+} (0.5 at.% Er^{3+}, 5 at.% Yb^{3+}).

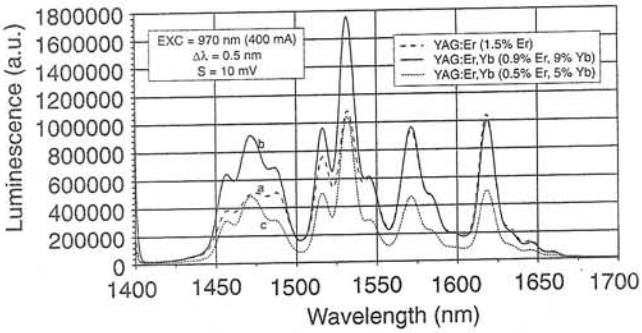

Fig. 5. Luminescence spectra of the erbium and ytterbium doped YAG crystals: (a) YAG:Er^{3+} (1.5% Er^{3+}), (b) YAG:Er^{3+},Yb^{3+} (0.9% Er^{3+}, 9% Yb^{3+}), (c) YAG:Er^{3+},Yb^{3+} (0.5% Er^{3+}, 5% Yb^{3+}).

Table. 2. Comparison of spectroscopic properties of Nd:YAG and Nd:YVO_4.

	Nd:YAG	Nd:YVO_4
Emission cross-section ($\times 10^{-19}$ cm^{-2})	2.8	15.6
Wavelength generation (nm)	1064.2	1064.3
Luminescence wide band (nm)	0.6	0.8
Lifetime for 1at% Nd concentration	230	100
Maximum of wavelength absorption	808	1808.5
Absorption cross-section ($\times 10^{-20}$ cm^{-2})	5	40.7
Absorption wide band (75% max) (nm)	2.5	15.7
Thermal conductivity (W/m/K)	10.3	5.14

tals were obtained by the flux technique what allowed to grow them at lower temperatures [5]. It decreased evaporation and changes of stoichiometry. Later it was found that the growth of YVO crystals is possible by the Czochralski technique and this was used in the presented work [6,7]. Good quality crystals containing 1–2 at.% of Nd were grown with the MSR puller equipped in RF-generator, and computerised regulation system connected with the Sartorius balance weighing crucible.

Measurements of a non-polarised absorption spectrum were made by means of Perkins Elmer Lambda Series PECSS spectrometer with 0.1 nm resolution in 750–850 nm range for the sample 0.2 mm thick made of 1 at% Nd doped YVO. The results are presented in Fig. 6.

Neodymium doped yttrium orthovanadate crystals due to high emission cross-section and linear polarisation output can find applications in many areas of laser technology, e.g., single frequency microchips, compact green lasers, OPO pumps, short pulse passively Q-switched lasers. Especially interesting for application are single frequency CW or passively Q-switched micro-lasers working at 1.3 μm and designed for high-transmission-rate fibre telecommunication.

$KGd(WO_4)_2$:Nd

Monoclinic $KGd(WO_4)_2$ (KGW), as many other double tungstates, reveals irreversible high temperature phase transition at 1278 K. Owing to this, growth of low-temperature phase of KGW, that have suitable lasing properties, has to be carried out from high temperature solutions. $K_2W_2O_7$ was used as a solvent. Two-zone resistance furnace allowed growing the KGW single crystals under the conditions of very low temperature gradients. Seeds were oriented in [010] direction. Growing crystals were rotated at 50 rpm and slowly pulled up at a rate of 2 mm/day [8]. As grown crystals had rectangular cross-section and flat (010) bottom. We have found by means of structural investigations that 10% content of Nd does not change the structure of KGW:Nd. Good quality KGW:Nd single crystals containing up to 8 at% of Nd were obtained. Figure 7 presents absorption spectra of KGW:Nd crystals. In Fig. 8, the luminescence spectra of KGW:Nd are presented.

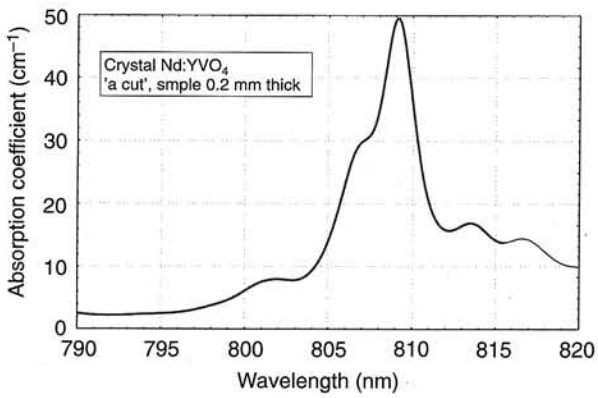

Fig. 6. Unpolarised absorption spectrum of elaborated vanadate crystal.

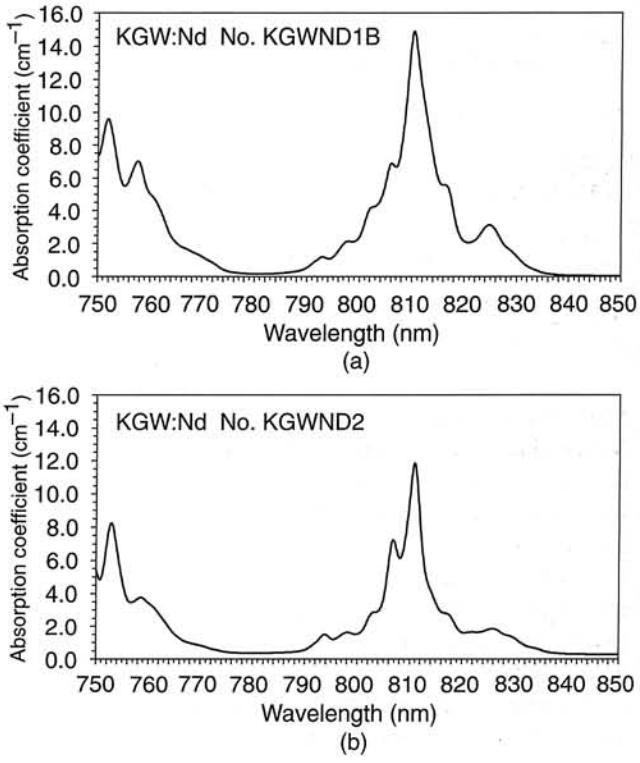

Fig. 7. Absorption spectra of neodymium doped KGW crystals: (a) KGW:Nd^{3+} (8 at.% Nd^{3+}), (b) KGW:Nd^{3+} (3 at.% Nd^{3+}).

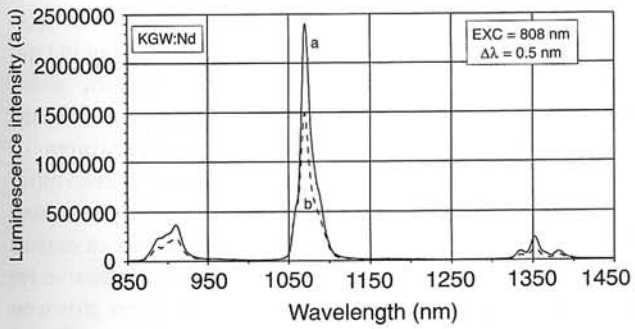

Fig. 8. Luminescence spectra of neodymium doped KGW crystals: (a) KGW:Nd^{3+} (8 at.% Nd^{3+}), (b) KGW:Nd^{3+} (3 at.% Nd^{3+}).

2.2. Self-frequency doubling crystals

In this subsection self-frequency doubling borate crystals are described. 1.06 μm laser oscillations are created and then doubled in the same crystals due to their NLO properties, what in a result gives 0.53 μm radiation.

$Ca_4GdO(BO_3)_3$:Nd

Gadolinium calcium oxoborate $Ca_4GdO(BO_3)_3$:Nd (GdCOB:Nd) is a new laser crystal possessing self-frequency doubling properties [9]. The main advantage of GdCOB, comparing with other borates, is relatively easy growth from stoichiometric melts. The material melts congruently at 1753 K, its viscosity is not very high, so the Czochralski technique (MSR-2 puller equipped in r.f. generator, pure nitrogen atmosphere) was used to obtain [010] GdCOB single crystals, pure and doped with neodymium up to 7 at% [10].

$YAl_3(BO_3)_4$:Nd (NYAB) crystals

NYAB crystals also combine NLO and lasing properties. NYAB melts incongruently, what imposes the necessity of using the HTSG technique. $K_2Mo_3O_{10}$ was used as a solvent. The crystal growth was carried out in a three-zone resistance furnace under low temperature gradients. Typical growth lasted 3–4 weeks, during which the temperature

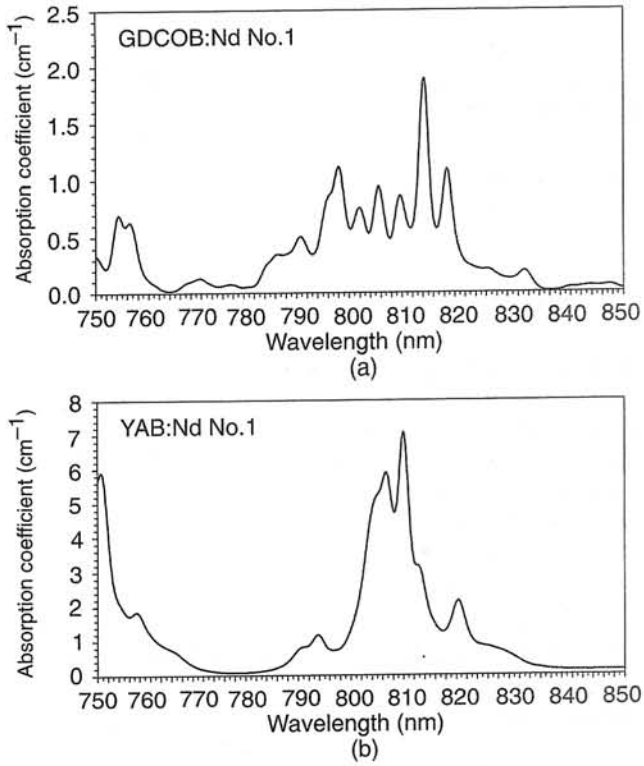

Fig. 9. Absorption spectra of neodymium doped borate crystals: (a) GdCOB:Nd^{3+} (3 at.% Nd^{3+}), (b) YAB:Nd^{3+} (5 at.% Nd^{3+}).

was lowered at a rate of 0.1 K/hr. No pulling was used. As-grown single crystals (1.5×1.5×1.0 cm^3) were confined with crystallographic planes and contained some inclusions. Figures 9 and 10 present absorption and luminescence spectra of GdCOB and YAB crystals activated with Nd^{3+} ions [11].

3. Nonlinear optical (NLO) materials for frequency conversion

Variety of optoelectronics applications imposes the necessity of generation of laser radiation having different parameters. NLO crystals are used in frequency conversion of lasers to obtain needed radiation length. The frequency con-

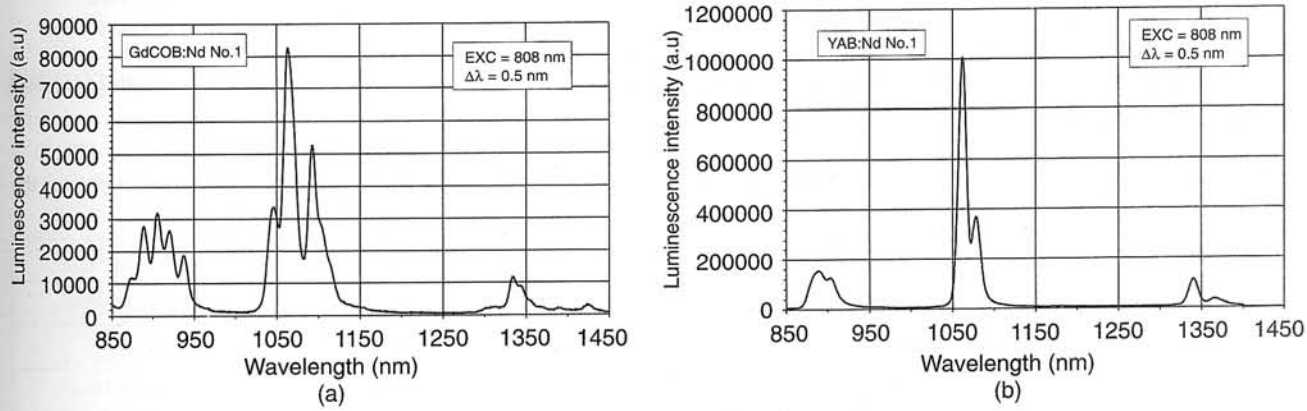

Fig. 10. Luminescence spectra of neodymium doped borate crystals: (a) GdCOB:Nd^{3+} (3 at.% Nd^{3+}), (b) YAB:Nd^{3+} (5 at.% Nd^{3+}).

version processes include second harmonic generation (SHG), which is a special case of sum frequency generation (SFG), differential frequency generation (DFG) and optical parametric generation. We have developed technology of several borate single crystals, which find applications in the above mentioned processes. In Table 3, BBO [12], CLBO [13], and LTB [14] single crystals for NLO applications are described.

4. Materials for Q-switched passive modulators

High power laser impulses having short lifetimes can be obtained in many ways by Q-switch modulation. One of the methods is passive modulation, which uses nonlinear absorption effect. During illumination of some materials one can observe the changes in transmission depending on the intensity of the radiation. When the laser radiation intensity reaches some level, the nonlinear absorbers become transparent and allow generating powerful impulses. Such passive modulators find many applications. We have investigated the Czochralski growth of several YAG doped crystals. Table 4 gives comparison of some spectroscopic parameters of these crystals [15,16].

5. Substrates for GaN epitaxy

For at least 30 years there has been a race to produce blue semiconductor laser diodes. Wide practical application for these kind of light-emitters makes large commercial market. The interest in optoelectronic devices seems to be growing within the II–VI and III–V compounds. Investigation results show that gallium nitride, GaN, looks very promising material for the blue electronic. However, growth of the bulk crystals of GaN that could be used as a substrate for homoepitaxy is rather difficult because of the decomposition of GaN, at about 1000°C at normal pressure. Growth of these crystals cannot be carried out in typical Czochralski configuration from stoichiometric melts. Growth is realised from the solution in liquid Ga under the pressur N_2 of 12–17 kbar at the temperatures of 1500–1700°C. Such high pressure is required to keep nitrogen within the growing GaN. The obtained crystals have the size of about 10 mm and exhibit high level of defects and not seem to be available commercially. Intensive research works are focused on GaN epitaxial layers grown on suitable substrates. Appropriate substrate for the epitaxial growth is crucial for practical application. The main factors determining the factors of adequate material used as a substrate are: crystallographic, chemical, physical, and also economical parameters. Lattice mismatch induces high concentration of defects. Large incompatibility of thermal expansion of GaN epitaxial layer and substrate causes cracks or peeling of the layer. Gallium nitride is hard material and has relativity small thermal expansion coefficient 5.6×10^{-6} K^{-1} while the most of oxides tend to have larger thermal expansion coefficients.

Table 5 lists some materials that are considered as potential substrates for GaN films. Presently, only sapphire is used as substrate in larger scale despite it has 16% lattice mismatch to GaN what gives high concentration of defects (10^7–10^{10}/cm^2) in GaN layers. Growth of $LiGaO_2$ and $LiAlO_2$ single crystals is connected with the same problems. During the growth changes in stoichiometry are observed and the obtained crystals are heavily defected.

Recently neodymium gallate is intensively investigated as a substrate for GaN epitaxy. The crystals are grown by the Czochralski technique and wafers of 2-inch in diameter are easily available. New and very promising material as a substrate for GaN epitaxial growth has been found, i.e., $(La,Sr)(Al,Ta)O_3$ (LSAT) that belongs to the cubic crystal

Table 3. Borates for NLO applications.

Material	Transparency (nm)	SHG coeff. (pm/V)	Damage threshold (GW/cm^2)	Method of crystallisation
β-BaB_2O_4 (BBO)	190–2500	2.3	5 (10 ns)	Flux Czochralski [12]
$CsLiB_6O_{10}$ (CLBO)	180–2750	0.95	26 (1 ns)	Self-flux Czochralski [13]
$Li_2B_4O_7$ (LTB)	200–3500	0.55	40 (10 ns)	Czochralski [14]

Table 4. Comparison of some spectroscopic parameters of nonlinear absorbers obtained by YAG doping; λ is the wavelength, n is the refraction coefficient, τ is the lifetime of the excited level, σ_a is the absorption cross-section.

Absorber	λ (μm)	n	τ	σ_a (cm^2)
YAG:Cr^{4+}	1.06	1.815	4 μs	2.5×10^{-18}
YAG:V^{3+}	1.06	1.815	5 ns	3×10^{-18}
YAG:V^{3+}	1.32	1.812	5 ns	7×10^{-18}
YAG:Co^{2+}	1.54	1.815	1 ns	9.2×10^{-19}

Table 5. Substrates for GaN epitaxy [17–20].

Material	GaN	Sapphire	$NdGaO_3$	$LiGaO_2$	$LiAlO_2$	LSAT
Space group	Hexagonal	Hexagonal	Orthorhombic	Orthorhombic	Tetragonal	Hexagonal
Mismatch	0	16	–1.3	0.1	–6.3(a); –1.4(c)	0.1
Thermal expansion $\chi \times 10^{-6}$ (K^{-1})	5.6(a) 7.7(c)	4.5(a) 5.3(c)	7.45	6(a) 9(b) 7(c)	7.1(a) 7.5(c)	—

system. It has very small lattice mismatch, below 0.1%. The crystals can be grown by the Czochralski method.

6. Materials for acousto-optic devices

Acousto-optic (AO) devices can modulate laser light, deflect it or change its frequency. Such applications as high-precision laser photoplotters, tunable AO filters, and display systems using 2-D beam scanning make AO materials wanted. We have developed the technology of TeO_2 single crystals grown by the Czochralski technique [21]. High-quality paratellurite single crystals enabled construction of AO devices having power consumption less than 0.5 W.

7. Conclusions

Owing to co-operation of IEMT, IAP and IO several single crystalline oxide materials have been obtained. Good quality single crystals of YAG with high concentration of neodymium, borates, vanadates, double tungstates, among others, create solid basis for the development of optoelectronics in Poland. The developed equipment as well as the experience gained during investigation of crystallisation of these materials can be used in future research of new crystalline materials that are still searched for.

Acknowledgements

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