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Blue laser diodes for trace matter detection

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We present spectroscopic applications of GaN-based laser diodes (LDs), fabricated and developed at the Institute of High Pressure Physics and its spin-off company TopGaN. LDs were manufactured using MOCVD technology. For spectroscopic applications we applied pulsed operated lasers generating the radiation in 395–430 nm spectral range reaching the power of 200 mW within the light pulses of 30–150 ns at the repetition rates of 10 kHz. These LDs were successfully applied to the detection of nitrogen dioxide in free atmosphere using cavity ring down spectroscopy (CRDS) sensor. Cw operated lasers generating within the spectral range of 385–420 nm and reaching powers up to 300 mW were adapted for atomic spectroscopy. Using external cavity tuning with diffraction grating in Littrow configuration, we obtained the stability of single mode generation better than 100 MHz within the periods of 30 minutes without any additional frequency stabilization feedback.

Keywords: trace matter detection, cavity ring down spectroscopy (CRDS), GaN laser diodes.

1. Introduction

Blue or near UV nitride-based semiconductor laser diodes (LDs) are nowadays commercially available. With regard to their short wavelength, lifetime and low power consumption they became suitable for applications such as fast laser printing, high density optical data storage and, recently, in pico-size projectors [1]. (Al, In)GaN based LDs also find many scientific applications, *e.g.*, in spectroscopy, where they can easily compete with expensive and large, standard spectroscopic sources (gas and solid-state lasers). However, spectroscopic requirements expect not only reliability of the laser device, stability of wavelength and power generation as well as good beam

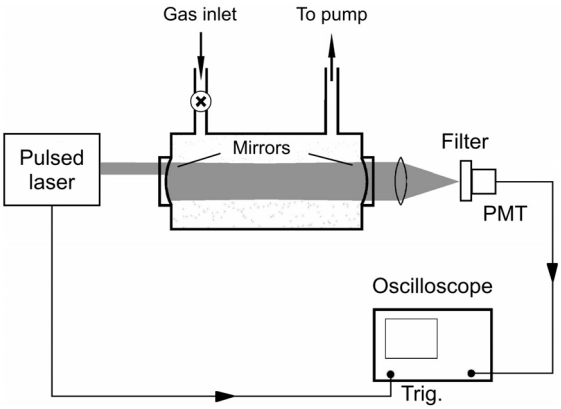

Fig. 1. Schematic view of typical CRDS experimental setup. Quality factor of the optical resonator can be determined measuring either the decay time of light inside the resonator [13], or the phase shift between input and output light pulses [19], or the intensity of light stored in the cavity [14].

quality. Equally important is the possibility to work with the laser in a broad, adjustable spectral range. Although semiconductor LDs can be specially designed to operate at given wavelength (varying doping and composition), broad spectral tunability is still an important feature of the device. The blue and UV spectral region (approximately 395–430 nm) – particularly interesting due to electronic absorption frequencies own by many atoms and molecules – became directly accessible with the development of GaN-based diodes. ZYBIN *et al*. [2] specified 34 chemical elements displaying strong absorption lines reachable with blue lasers. Precise atomic spectroscopy employing this source of light was already demonstrated for indium [3,4], rubidium [5], potassium [6] and calcium [7]. Among the compounds which are commonly present in the atmosphere, nitrogen dioxide has the maximum of blue–UV absorption bands in the 370–440 nm spectral range. Based on this feature, a construction of a highly sensitive sensor of the NO_2 compound has already been reported [8–12]. With regard to the above mentioned facts blue LDs are conveniently applicable to detection of the lowest concentrations of various atoms and molecules, where the principle of trace matter detection relays on small absorption measurement. Cavity ring-down spectroscopy (CRDS) and its modifications are among the most suitable techniques for such investigations [13–17]. Applying these methods (see Fig. 1), the laser pulse is trapped inside the optical resonator of a very high quality (Q). When the laser wavelength matches the absorption line of the matter filling the resonator, this quality factor Q decreases. The absorption coefficient can be then found comparing the quality factors characterizing the empty resonator and the resonator filled with the absorber. We can use pulsed operated lasers as the light sources as well as cw lasers, amplitude modulated. It was already demonstrated that for pulsed lasers the sensitivity of the absorption coefficient determination can reach 10^{-9} m^{-1} while for cw lasers the measurement of the absorption of 10^{-12} m^{-1} is possible [18].

2. Pulsed blue lasers for trace matter detection

The pulsed laser diodes were constructed at the Institute of High Pressure Physics (Warsaw, Poland) in collaboration with its spin-off company, TopGaN. In Figure 2 we show a schematic view of the laser diode design. The epitaxial structure was fabricated using metalorganic chemical vapor phase epitaxy (MOVPE) vertical flow reactor on the high pressure grown gallium nitride substrates [20]. The initial density of dislocation in the substrate material was below 100 cm^{-2}. The active region consisted of 3–5 undoped quantum wells (3–4 nm thick), separated by 8–15 nm, silicon-doped $In_{0.09}Ga_{0.91}N$ barriers. The electron blocking (EBL) was formed by a 200 Å thick Mg-doped $Al_{0.2}Ga_{0.8}N$ layer. The $Al_{0.08}Ga_{0.92}N$ cladding layers (0.35 μm and 0.55 μm for upper and lower layer) sandwiched 0.1 μm GaN waveguides. The final density of dislocation in the structure, as measured by the selective etching method, was between 5×10^4 cm^{-2} and 1×10^5 cm^{-2} [21], which means just a few dislocations per laser stripe. The chips of the dimensions of 300 μm × 500 μm were mounted *p*-side down on a diamond submount and packaged in a standard 5.6 μm TO can.

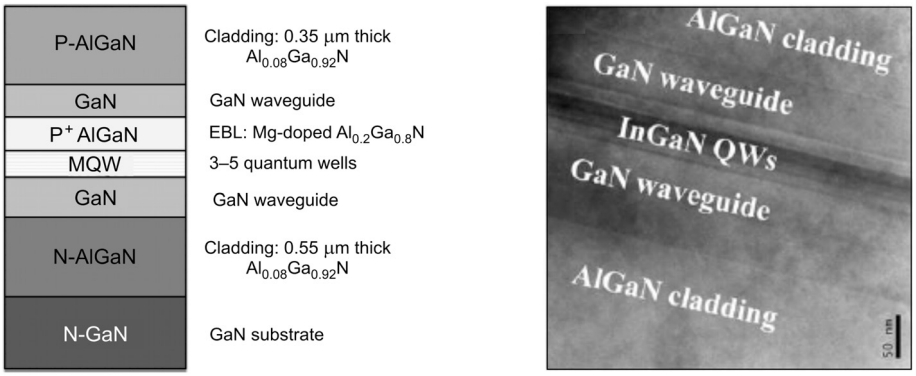

Fig. 2. LD epitaxial structure: schematic layer design (**a**), TEM image of the grown sample (**b**).

For the highly sensitive detection of trace elements the essential laser parameter is the maximum optical power (peak power) of the laser. To achieve this goal, the slope efficiency of the device needs to be optimized because this parameter has a crucial influence on the laser output power. Simultaneously, we must pay particular attention not to exceed operating current densities limited to values at which catastrophic optical mirror damage (COMD) occurs. Finally, the possibility of effective coupling of the laser beam with external cavity (CRDS) is determined by the beam profile so we must also have in mind proper beam shaping.

In order to fabricate nitride laser diodes with high differential efficiency, it is necessary to minimize the optical losses of the resonator. The main source of the optical losses is the presence in the laser structure of layers highly doped with magnesium and

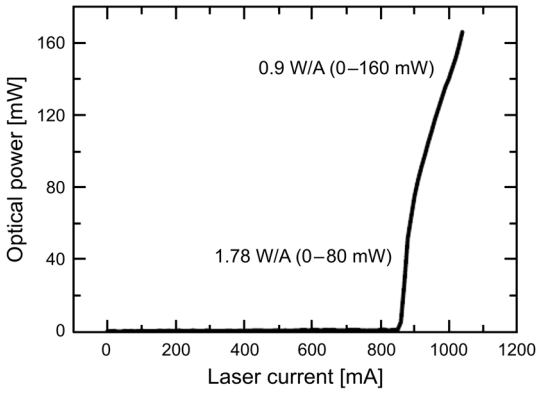

Fig. 3. Light-current characteristics of the laser structure with strongly asymmetric waveguide.

Mg-related/induced deep centers. We introduced the special design of the resonator with strongly asymmetric waveguide for which the overlap of the transversal mode with the Mg-doped regions is minimal. Owing to this, we were able to realize devices with slope efficiencies reaching 1.8 W/A, as it is shown in Fig. 3.

In case of nitride LDs, the COMD limit value is around 40–50 MW/cm^2, where the indicated density refers to the density of the optical field on the laser facets. In order to overcome the mentioned barrier, we introduced wide-stripe design to our lasers. Figure 4 demonstrates the parameters of 50 μm laser diode which reaches the output power of 2.5 W.

Finally, discussing the beam properties of the nitride lasers one has to mention the problem of the electromagnetic mode leakage into substrate [22]. This leakage, besides its detrimental influence on the optical confinement coefficient of the laser, leads to drastic worsening of the near and far field patterns. In order to suppress this leakage, we introduced a new concept of the plasmonic cladding [23] which practically eliminated this effect.

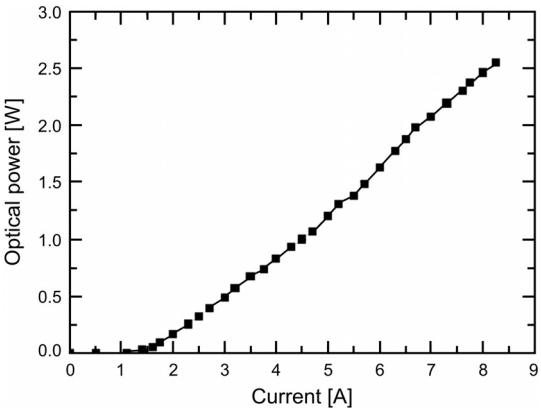

Fig. 4. Light-current characteristics of the laser with 50 μm wide stripe.

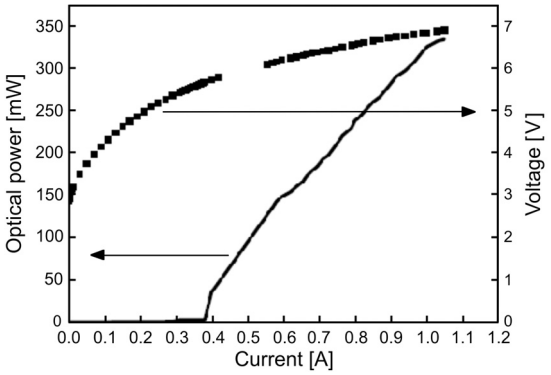

Fig. 5. Light-current and voltage-current characteristics for our high power LDs. The ridge size is 10 μm × 700 μm.

The maximal output powers achieved with our devices were 340 mW cw (see Fig. 5) in case of ridges size of 10 μm × 700 μm. In those cases we used a specially designed copper mounting providing proper thermal management of the device. For spectroscopic applications we use lasers with narrower ridges (3 μm) in a standard TO-can mounting and typical power of around 5 mW.

3. NO_2 detection with pulsed diode lasers

NO_2 occurs in the air due to various anthropogenic processes as well as due to industrial activity, fuel burning *etc*. It is the key factor that characterizes the quality of the atmosphere and for this reason it is usually monitored. Nowadays, when the security problems attain more and more significance, the sensitive detection of nitrogen dioxide becomes particularly important. NO_x compounds usually appear during decomposition of many military explosives, and therefore they can be used as an indication of the presence of dangerous materials. Currently NO_2 is commonly detected using the methods based on chemiluminescence [24, 25]. Their sensitivity reaches single ppb. As mentioned in the Introduction, the emission range of blue diodes (covering wavelengths in the range of 395–430 nm) fits well to NO_2 absorption spectrum. Within these wavelengths nitrogen dioxide has $^2A_1$–$^2B_1$ and $^2A_1$–$^2B_2$ absorption bands with mean cross section of 6×10^{-19} cm^{-2}, varying relatively slightly – only about ±40% (Fig. 6). Unlike in case of atomic spectroscopy, which we describe in the next section, we can apply any diode lasing in this area. In addition, among common atmospheric gases there is no other compound that absorbs in this spectral range. Therefore pulsed violet and blue diode lasers with application of CRDS techniques (and their modifications) might lead to construction of fully optoelectronic sensor of this compound. First laboratory constructions have been demonstrated already [8, 9, 12, 26, 27].

Pulsed lasers usually are not tunable and their linewidth is of the order of 1 nm. As far as they are not very suitable for atomic spectroscopy, almost every blue diode

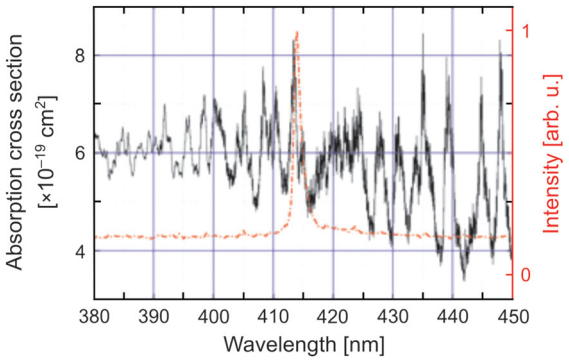

Fig. 6. Absorption cross section of NO_2 in the spectral range corresponding to blue LDs emission [28] and the emission line profile of the laser that was applied in our experiment (dashed line).

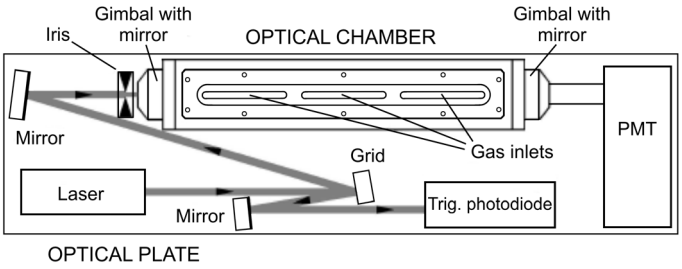

Fig. 7. Schematic view of our NO_2 sensor.

is useful for NO_2 detection due to properties of the absorption spectrum. Accurate control of their supply current results in generation of rectangular light pulses of 50–100 ns duration time at the repetition rate reaching several kHz, which conveniently suits CRDS technique.

Construction of our NO_2 sensor is shown in Fig. 7. It consists of a chamber that is winded to an optical table. The cavity mirrors are mounted in two gimbals (Los Gatos) which are fixed to frontal surfaces of the chamber. The gimbals seal up the mirrors to the chamber and ensure precise regulation of their tilt over the angle of ±3° in respect to the cavity axis.

We applied blue laser diode working around 414 nm as the light source. It was operated in the pulsed regime generating pulses of peak power about 200 mW, duration time 100 ns and the repetition rate about 10 kHz. The laser beam illuminated a blazed grating (density of 2400 mm^{-1}). The beam reflected at the 0th diffraction order was directed to a photodiode to produce the reference signal. The beam deflected at the 1st diffraction order passed through an iris diaphragm and illuminated the cavity. The use of the grating and diaphragm eliminates the broadband electroluminescence of the laser diode. The spectrum of this luminescence is much broader than the width of high reflectivity band of the cavity mirrors and might strongly affect the shape of the measured exponential decay signal. Applying the iris diaphragm also helps to avoid

the multimode excitation of the cavity, which may eventually lead to multiexponential decay of trapped radiation and cannot be interpreted without doubt.

Injecting light into the cavity we applied off-axis approach (see Fig. 1) which allows the laser beam to reflect hundreds of times inside the cavity without overlapping [29, 30]. In this way we avoided closely spaced longitudinal cavity resonances (usually present in the on-axis excitation method). In such case the frequency spectrum of the cavity is characterized by dense mode structure with weak and broad longitudinal modes. The cavity couples well to broadband and unstable laser radiation and does not need any additional system of cavity length control or laser frequency tuning. Finally with this arrangement the back reflected laser beam by a convex surface of the front mirror does not reach the diode laser and does not disturb its work. The cavity was formed by two mirrors (Los Gatos) with reflectivity reaching beyond 0.9999 at the wavelength of 405 nm. The distance between the mirrors was 37 cm and the radii of curvature were 1 m for both mirrors. To determine the cavity Q factor, which is necessary for measurements of NO_2 concentration, we performed measurements of the radiation trapping time. Then, we determined the absorption coefficient using the following relation

$$\alpha = \frac{1}{c}\left(\frac{1}{\tau} - \frac{1}{\tau_0}\right)$$

In case of an empty resonator, we observed the decay time of $\tau_0 \approx 8$ μs. During the measurement the cavity was supplied with a gas (NO_2–air) mixture of 50 ppm mixing ratio. The mixture was additionally diluted in pure nitrogen using two gas flow controllers (Beta-Erg) so that we could regulate the mixing ratio of the gas inside the cavity. To avoid disturbing homogeneity of the medium in the cavity, we kept the gas flow reduced to 3 l/min. The output signal was recorded with a fast 8-bit digital oscilloscope (HP 54520) and a lock-in amplifier.

In Figure 8 we demonstrate the results of our investigations. There is a good agreement between the measured absorption coefficients and the corresponding NO_2

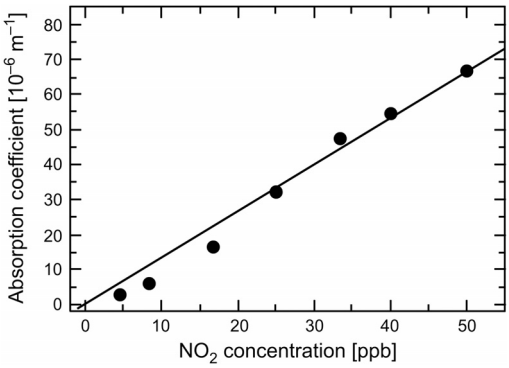

Fig. 8. Measured absorption coefficient of NO_2 versus the concentration of prepared mixture.

concentrations when the absorption cross section of 5×10^{-19} cm^{-2} is assumed. On the other hand, for the wavelength of our laser (414 nm) the value of this cross section is 5.5×10^{-19} cm^{-2}. The error can occur not only due to CRDS technique uncertainty but also due to the error of the mixture preparation. The uncertainty of linearity coefficient does not exceed 5%. The sensitivity of single ppb, which we achieved in measurements presented here, can be further increased by application of specialized electronics improving the precision of measurement of the radiation decay time.

4. The outlook: CW blue lasers for matter detection

Unlike in case of molecular absorption measurements for high precision atomic spectroscopy, where the operation with precise wavelength is required, the tuning and spectral characteristics of free running laser diodes are far from ideal. A solution to the problem comes with the use of external cavity systems with addition of antireflection (AR) coatings applied on the LDs facets. Coupling the laser to external resonator affects the emission spectrum of the diode and also introduces modification to the threshold current and the output power [31–33]. Single mode operation and tunability of the laser line over the range of the diode medium can be obtained with the introduction of frequency selective feedback, typically through diffraction gratings in either Littrow [34] or Littman [35] configurations. In the latter configuration, which is more frequently employed, the first order of diffraction is fed back directly into the laser diode while the specular reflection from the grating (0th order) forms the output beam. Tuning of the wavelength is obtained by rotation of the grating, however along with the change of wavelength we also alter the output beam direction. This drawback can be solved by the use of an additional mirror mounted with the diffraction grating on a common base, allowing to rotate the both elements simultaneously [36].

Using our broad ridge (Al, In)GaN cw LDs in the external cavity we obtained promising results in terms of their applicability as light sources to atomic spectroscopy. The epitaxial structure was very similar to that described in the previous paragraph.

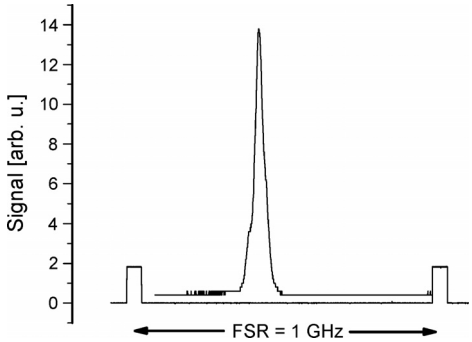

Fig. 9. Single mode emission of cw-operated InGaN blue LD. Rectangular line represents free spectral range markers of Fabry–Perot interferometer ($\Delta\nu$ = 1 GHz).

The LD was operating at 405 nm, the output power was 5 mW. Using Littrow configuration and providing temperature stability of 0.01 °C we obtained narrow, single line emission. This observation is presented in Fig. 9. The registration was done with 1 GHz Fabry–Perot interferometer. The output intensity was registered by a photodiode and digital oscilloscope. Diffraction grating of 3600 l/mm formed the external resonator of a length of 5 cm. Stability of generation was better than 100 MHz within the time periods of 30 min without any active feedback system.

5. Conclusions

We demonstrated the applicability of nitride laser diodes to trace element detection. By combining our pulse laser diodes with CRDS system, we were able to reach the detection level of single ppb for NO_2 detection. We also applied the external cavity configuration (Littrow) to obtain spectrally narrow and single mode emission from our laser diode. We achieved the spectral stability and width of the emission close to 100 MHz for periods of several minutes without using any feed-back loop laser frequency control. This is sufficient for the atomic spectroscopy at Doppler broadened lines which usually are characterized by widths of GHz. A spectral range of the presented lasers makes them useful for experiments for many trace elements such as indium, gallium, *etc*. [2]. Huge oscillator strengths of atomic transitions, typically 3–7 orders of magnitude higher than that for molecules, provide an opportunity of even single atoms/cm^3 detection when high sensitivity spectroscopy techniques, like CRDS, are employed.

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