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THESIS BOOKLET

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Bandwidth widening of LEDs for near-infrared spectroscopy and lighting applications

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1. Background to the research

LEDs have undergone tremendous development in the last two decades, becoming the dominant light source in lighting technology, bringing a revolutionary change among artificial lighting devices that can perhaps only be compared to the appearance of the incandescent lamp. From the replacement of household light bulbs with retrofit LED lamps, through various public building lighting fixtures to special sports and decorative lighting devices, they have replaced previous traditional light sources everywhere. The same happened in the automotive industry, in the case of light sources serving as backlights for displays, TVs and in many other specialized fields.

During the same period – due to, among other things, the further miniaturization of electronic devices and printed circuits and the increasing energy density of batteries – the demand for the development of small, lightweight, portable devices in the field of industrial and laboratory optical instruments increased, which required the replacement of the previous incandescent lamps or other built-in light sources with higher power requirements with smaller, electrically and spectrally optimized, special LED light sources. Instrumentation became one of the earliest and also the latest lighting application areas to change light sources. Some optical instruments require light sources (radiation sources) emitting in the invisible range, which were already available before the widespread use of LEDs for lighting purposes, but many instruments require broadband LEDs that are not yet available or have only been available for a few years.

The development of LED light sources is a continuously and dynamically developing sub-area of lighting technology, where instead of further increasing the efficiency of LEDs, the development of their spectral quality parameters is increasingly being prioritized.

LEDs emit in a narrow spectrum, which is their biggest advantage in terms of energy saving, as they emit energy exactly where it is needed and LEDs can be made to the "color" (wavelength) that is needed by selecting the material composition. This is a huge advantage compared to the unnecessary radiation losses of incandescent lamps that emit in a wide spectrum and discharge lamps that require wavelength conversion**. However, this is also the biggest disadvantage of LEDs:**

for most applications, including everyday lighting, this band is too narrow. In lighting technology, the blue LED + yellow phosphor solution dominates, this is a well-known way of band broadening, but the phosphor wavelength conversion also involves significant losses in LEDs and its color quality needs further development. **The focus of LED research worldwide is efficient bandwidth widening.**

My work has brought me into contact with one of these important applications, near infrared (NIR) spectroscopy. **GaInAsP is a semiconductor with a wavelength that is practically tunable in the whole NIR range, so I focused on it.** The goal is to develop a more accurate and efficient bandwidth widening method than light-powder bandwidth widening, which is more useful for instrumental measurements. **The main focus was on the applicability in handheld applications, i.e. low power consumption, small size, true point-like characteristics despite multiple emission wavelengths, and temperature independence to avoid tempering energy losses.**

A significant part of the research focused on the novel structure, operation, and growth of the NIR LED, so I will present it in more detail in my thesis. **I would like to contrast the NIR LED band widening solution with similar band widening problem lighting LEDs, from my research to increase visual comfort.** I have extended the bandwidth broadening studies to lighting technology: **the spectral characteristics and functional adequacy of hybrid LEDs with low luminous efficacy white LEDs and red LEDs in different colour rendering schemes have been investigated.**

In both cases, I wanted to work in a practical, results-oriented way. For the NIR range, I wanted **to make an LED that had never been made before in the GaInAsP material composition** and has the potential to be a product later. And for the VIS research, I wanted to achieve a result that would contribute to improving our everyday visual environment, such as home lighting. **The results achieved in each sub-area can be used for other LED applications**: e.g. from the sub-areas of human-centred lighting or plant lighting to the further development of LEDs for instrumentation and vice versa.

2. Objectives

The aim is to investigate the structure of GaInAsP/InP compound semiconductor layers as a NIR spectroscopic handheld LED light source for organic materials and biological samples. In summary:

- **Layer structure suitable for a single chip (not diode array).**
- **Its peak wavelength can tune over the full NIR II range.**
- **Contains a bandwidth widening solution, but not as a post-applied phosphor layer.**
- **In order to maintain precision, the optical axis dimension of the band broadening layer does not exceed the size of the light-emitting surface.**
- **The wavelength range covered by the bandwidth widening should cover the wavelengths of the harmonics of at least two functional groups, preferably the wavelengths of the OH and CH harmonics.**
- **A high-efficiency, low-power, small-sized chip should be produced using this method.**
- **Its spectrum should be nearly temperature independent in the measurement wavelength range.**
- **Its spectrum should be nearly direction-independent in the measurement angle range.**

In the case of the lighting sub-area, the goal is to expand bandwidth widening in order to reduce the use and loss of phosphor:

- **Implementation of a hybrid LED light source and luminaire using red LEDs and white LEDs with minimal phosphor content.**
- **Examination of white LED bandwidth widening based on luminous efficacy and colour rendering, with optimization of component characteristics.**
- **Evaluation of the interplay between phosphor usage, efficiency, and colour quality.**

3. Scientific methods

The experimental LEDs were grown using the liquid-phase epitaxy (LPE) method. The reactor tube of the LPE equipment was made of quartz and heated by a circular, specially wound self-supporting Kanthal spiral. The furnace (or kiln), which concentrically surrounded the reactor tube, was also made of quartz and had a thin gold layer vapor-deposited on the inner surface using the PVD method. This gold layer's primary purpose was to reflect heat. The boat within the reactor chamber was made of graphite, with a replaceable section designed to hold the melts, adaptable to the required number of cells. The stator also had an interchangeable socket, allowing the accommodation of substrates of varying sizes. Growths were conducted under H_2 protective gas. The temperature was monitored using two Pt/Pt-Rh thermocouples directly connected to the control unit. The control system featured a programmable electronic actuator and a temperature controller.

The experiments focused on the growth and analysis of a GaInAsP/InP structure that, in addition to the standard layers typical of this material system, included a luminescent layer. According to the hypothesis, the luminescent layer absorbs part of the radiation from the active layer and re-emits it at a longer wavelength, enabling the LED to produce multiple emission peaks simultaneously. The wavelengths of the emission peaks can be adjusted by modifying the composition of the compound semiconductor layers, while their intensities can be controlled by varying the thickness of the layers.

During the design phase, I determined lattice-matched components corresponding to the selected wavelengths and the associated bandgaps. Based on these, I calculated the solid-phase compositions. Polynomial functions were fitted to the solubility distribution coefficients, allowing the calculation of mole fractions in the liquid phase. From these mole fractions, the required quantities of Ga, As, and P were derived and pre-calculated.

Additionally, I determined the temperature-time data pairs associated with each layer. The quantities to be measured and the growth parameters were documented in tables. LEDs resulting from individual growths were assigned unique serial numbers for identification in subsequent analyses.

The experimental LEDs, in line with the application area for handheld instruments, were 300×300 μm² chips with a current of 50 mA. The peak wavelengths of the radiation from the active and luminescent layers were verified on the unpackaged LEDs using spectral transmission measurements with a NIR spectroradiometer. Since the LED is passive during transmission measurements and does not heat up, more accurate values can be obtained compared to emission measurements in the active state. The emission peaks were identified as local maxima in the first derivative of the spectral transmission curve, and with tolerable variation, they matched the planned wavelengths.

The intensities and intensity ratios of the emission peaks were examined using spectral emission measurements, with DC power supply, a needle measurement microscope, and an NIR spectroradiometer. The emission measurements confirmed that the integration of the luminescent layer was a functional design. The magnitudes of the two emission peaks and their intensity ratio were in line with the planned values, within a tolerable margin of error.

For the mobile spectroscopic application, temperature-dependent measurements were performed at a constant current, with varying temperature, using spectral intensity measurements. These were metal-packaged LEDs, and temperature measurements were conducted on the package. The input data required for the theoretical model experiment were obtained from temperature-dependent intensity measurements of LEDs without a luminescent layer (with a single emission peak). The LEDs designed with the model, featuring two emission peaks, exhibited a nearly temperature-independent section.

The LEDs, grown based on the models, were checked using spectral intensity measurements at temperatures ranging from 30–100°C. The nearly temperature-independent sections formed between the two emission peaks when the distance between the peaks was less than 110 nm, where the temperature-dependent relative intensity change was even smaller than expected. For emission peaks that were further apart, nearly temperature-independent sections were formed between 30–50°C.

Due to the potential inaccuracies in the direction of manual measurements, I also examined the spectral angular dependence of the luminescent LED within a ±20° range from the optical axis. The measurements were performed using an NIR spectroradiometer under stabilized current and temperature parameters. The luminescent layer slightly enhanced the spectral angular dependence of the radiation paths originating from the active layer (and passing through the luminescent layer), but the effect remained low. The angular dependence of the radiation generated in the luminescent layer was negligible. Overall, the integrated luminescent layer did not affect the spectral angular dependence of the LED to a degree that would threaten its intended application.

To ensure the long-term operation of the LED, its radiation intensity is stabilized, allowing for variations in the driving current. This is particularly critical for compensating for intensity degradation due to aging. Consequently, I needed to investigate the extent of spectral intensity changes associated with variations in the driving current. These measurements were performed with the LED package maintained at constant room temperature using Peltier element-based temperature stabilization. To minimize the temperature difference between the package and the active layer, the LED was operated in QCW mode powered by a PWM power supply with a constant duty cycle of 1/1000. According to the spectral measurements, this method caused no measurable heating in the active layer up to twice the nominal current, allowing spectral intensity measurements within this range. For currents below the nominal value and up to twice the nominal current, the spectral intensity variations between the two emission peaks were insignificant, provided the wavelength of the active layer did not exceed 1300 nm.

To demonstrate the practical applicability of the luminescent GaInAsP LED for spectroscopic purposes, I performed a measurement model experiment. The objective was to detect ethanol in water. Ethanol serves as a good model substance, effectively representing the measurement of organic compounds in practical applications. In a previous patented method, the ethanol-water mixture was typically analysed using three separate LEDs at three different wavelengths. I confirmed that the dual-wavelength emission of the luminescent GaInAsP LED, combined with a sandwich detector, enables concentration measurements in the ethanol-water system with the accuracy expected from handheld instruments.

In GaInAsP compound semiconductors with higher Ga content, there is a significant tendency for re-dissolution into the InP melt. To prevent this, an anti-dissolution layer is grown before the luminescent layer. This layer is designable and tuneable in terms of thickness and composition, allowing it to function as an absorbing layer. Electrons released within the absorbing layer can migrate only in one direction, toward the luminescent layer. This results in an energy conversion process that is theoretically 100% efficient, although quantum losses due to wavelength differences are inevitable. Within the scope of the experiment, I modelled the internal conversion efficiency of the luminescent layer and measured it using spectral emission. The measurements showed that this efficiency was close to 100%, significantly outperforming the conversion efficiency of the well-known phosphor-based bandwidth widening method used in LEDs.

In the case of LEDs grown with absorption and luminescent layers, the compliance of layer thicknesses and compositions was verified using X EDS SEM (X-ray energy dispersive spectroscopy, scanning electron microscopy) measurements. The flawless operation of the active layer was confirmed through EBIC (electron beam induced current) measurements. During the investigations, there was also the opportunity to detect growth anomalies and defects. The tuning of the compositions, layer thicknesses, and peak wavelengths was done within the expected, low manufacturing variation. The measured results were consistent with the planned values.

The use of luminescent layers in lighting technology is limited due to the different material compositions of LEDs for lighting purposes and the 400 nm spectral range of human vision. To address this, I extended my work with an experiment in the visible range that complements the spectrum of white LEDs with poor colour rendering properties and relatively low amounts of phosphor by adding a red LED. In this case, the bandwidth widening is partially provided by a separate LED from the white LED, and together they form a hybrid LED. I investigated how much the radiation efficiency of the hybrid LED decreased compared to the white LED when varying amounts of red LED light were added. The experiments involved six combinations of three white LEDs with different colour temperatures and two red LEDs with different peak wavelengths. I explored which combination and the optimal proportion of red LED light would yield the best results. I also investigated the reduction in radiative efficiency that this combination caused. For the measurements, I used DC power supply, an Ulbricht sphere, and a spectroradiometer operating in the VIS range. The uniqueness of the experiment lies in the fact that, after the measurements, I calculated the colour rendering values using CRI, CQS, and TM-30 colour rendering systems and compared them.

I created demonstration light luminaires from the above LED combinations. In contrast to the previous experiment, here I specifically examined the change in relative photometric light efficiency. One of the variables was the different proportions of red LED light mixed with the white light, while the other two variables were the currents of the white and red LEDs. The result of the investigation was the identification of the optimal red-to-white light ratio. At this ratio, the hybrid LED provided the best performance in any of the colour rendering systems tested, with the smallest reduction in light efficiency.

4. New scientific results

1. **I demonstrated that in the GaInAsP/InP material system, it is possible to create a LED with a layer structure in which, apart from the active layer, a secondary quaternary layer (luminescent layer) absorbs part of the primary radiation and re-emits it at a longer wavelength. This enables a single LED to emit simultaneously at two closely spaced near-infrared wavelengths. The outstanding significance of this achievement lies in the fact that it has not been realized previously in this material system.** The wavelengths can be adjusted by designing the composition of the quaternary layers, while the intensity ratios of the peaks can be tuned by designing the layer thicknesses. The alignment of the wavelengths with the design specifications was verified through transmission and emission studies, with an average deviation of $\Delta\lambda_{av}$ = 4 nm. [S1] [S2] [S3] [S4] [S6] [S9] [S12]

2. **Through experiments, I demonstrated that the GaInAsP/InP material composition LED, created with a luminescent layer, emits at two wavelengths and is suitable as a temperature-independent radiation source in the near-infrared range.** For nearby (less than 110 nm) peak wavelengths, a region with minimal temperature dependence (with a relative intensity change of at most 2%, remaining nearly temperature-independent within practical temperature ranges) forms between the peaks. The width of this region matches the distance between the two peaks. For more distant (greater than 110 nm) peaks, a relatively wide regions with minimal temperature dependence forms around the higher wavelength boundaries of the half-widths. These regions are at least a quarter of the half-width in size. Within these regions, the radiation remains nearly temperature-independent, with a relative intensity change of at most 2%, under the practical temperature limits.. [S2] [S3] [S4] [S5] [S6] [S7] [S9]

3. **I have determined that the luminescent layer of the GaInAsP/InP compound semiconductor LED, which radiates**

at two wavelengths, hardly alters the spatial spectral intensity dependence within a ±20° angular range relative to the optical axis. From this perspective, the LED is suitable for NIR spectroscopic applications. In the angular range of $\Delta\beta \leq \pm 20°$ relative to the optical axis, the directional dependence of the luminescent layer is negligible (within measurement uncertainty), and the spectral intensity dependence of the active layer deteriorates only slightly compared to a similar LED designed for a different wavelength, but without the luminescent layer. The spectral intensity dependence of the active layer, due to the incorporation of the luminescent layer, showed a maximum relative intensity variation of 9% at the most distant angle (20°) from the optical axis. [S5]

4. **I have proven that a GaInAsP/InP compound luminescent LED, at room temperature and for peak wavelengths smaller than 1300 nm (under conditions suitable for handheld spectroscopy), when driven up to at most twice of the nominal current, exhibits nearly current-independent spectral radiation and relative intensity.** The GaInAsP/InP compound semiconductor LED with two peak wavelengths, driven in DC mode between the nominal current and its double, shows a relative intensity variation of no more than 1.5% between the two peak wavelengths, measured at any wavelength between the peak wavelengths. [S5]

5. **I have demonstrated that by increasing the thickness of the passivation layer and tuning its composition to the desired wavelength, an absorbing layer can be created between the active layer and the luminescent layer of the GaInAsP/InP compound semiconductor LED, which generates electrons for the luminescent layer from the primary radiation of the active layer. The significance of this result lies in the fact that this had not been previously achieved in this material system.** For LEDs made with such layer structures, I confirmed the compliance of the designed wavelengths through transmission and emission tests,

with the average deviation from the planned values being $\Delta\lambda_{av}$ = 1.25 nm. [S2] [S3] [S4] [S8]

6. **I have confirmed through measurements that the spectra of the GaInAsP/InP LEDs, which emit at two wavelengths with a luminescent and complementary absorbing layer, match the planned specifications. Additionally, I have verified that the peak wavelengths, layer structures, compositions, and thicknesses of these LEDs also align with the design specifications. Therefore, the design methodology developed for such composite layer systems in this material system is appropriate.** The deviation of the measured layer thicknesses from the planned values was within 1% for the most accurately fabricated LEDs, and between 5–10% for the least accurate sample. [S5] [S8]

7. **Through experiments, I have demonstrated that using a neutral white LED (Tk > 5000 K) with low light scattering and weak color rendering (Rf < 75), along with a red LED (λ_p = 640 nm), an ideal warm white hybrid LED light source and, in this way, a luminaire suitable for general lighting purposes can be realized ($R_f \geq 85$, $R_g \geq 108$).** This bandwidth widening method reduces the use of phosphor by one third at the cost of a 13% decrease in radiative and photometric efficiency [S10] [S11]

5. Possibility of exploiting the results

Through precise component design and liquid-phase epitaxy (LPE) growth, a GaInAsP/InP compound semiconductor LED was successfully created that emits light at two wavelengths simultaneously, as part of the active layer's radiation is converted by a luminescent layer into light of a different wavelength. The significance of this achievement lies in the fact that such a structure had not been previously realized in this material system. The emission peaks can be precisely designed with the compositions, and the relative amplitudes with the layer thicknesses. The resulting LED is suitable for spectroscopy measurements as a single light source.

Thanks to the pinpoint metallization, light is generated in a concentrated manner at high current density within a narrow channel, making the light source itself extremely point-like, with light emission occurring over an area on the order of 100 µm. In the narrow range (±20°), the emitted radiation spectrum is virtually independent of direction.

In the wavelength range between the two peaks, the relative spectral emission is practically independent of temperature and current under reasonable usage conditions.

In order to prevent back dissolution, a back-dissolution-preventing layer needs to be grown. By growing this layer with the designed composition and thickness, an absorption layer is formed, which generates charge carriers for the luminescent layer from the primary radiation of the active layer. Such a layered structure has not been created before in this material system. The emission peaks can be precisely designed with the compositions, and the relative amplitudes can be adjusted with the layer thicknesses.

The internal efficiency of the wavelength conversion of the luminescent layer is nearly 100%, which significantly exceeds that of phosphor-based solutions.

As a result of this work, the developed LED is suitable for NIR spectroscopic measurements in two wavelength bands or between nearby peaks as a single point source, greatly simplifying and making the measurement more accurate. Due to its small size and power consumption, as well as its temperature-independent characteristics and the possibility of eliminating the need for temperature regulation, it is especially suitable as a light source for mobile devices.

The design was patented, but the maintenance of the patent was discontinued due to financial reasons. The GaInAsP/InP LED, which radiates at two wavelengths, is available as a product at the research institute, where it can be ordered with customized specifications (wavelength and other parameters).

The layer structure, thicknesses, compositions, and functionality were in accordance with the design. I validated this through X EDS SEM and EBIC analysis of these layers, thereby proving the correctness of the method used for designing the absorbing and luminescent layer system.

The two-wavelength GaInAsP LED design developed in this way is suitable for further development in metrological applications, particularly for NIR spectroscopy in handheld instruments. Additionally, the bandwidth widening method could, in principle, be applicable for the development of LEDs for lighting purposes, potentially replacing part of the phosphor content.

I conducted investigations on partial phosphor replacement and bandwidth widening in the visible range as well. The requirements for warm white LEDs in lighting technology can be met using a hybrid LED made from neutral white LED and red LED. This bandwidth widening method reduces phosphor usage by one third at the cost of a 13% decrease in radiometric and photometric efficiency, while maintaining color rendering and saturation values that far exceed the expected values for both the light source and the lighting fixture made from it.

6. Own scientific publications related to the thesis points

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