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Dissertation Thesis Abstract

Electrical and optical characterization of progressive light-emitting diodes

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INTRODUCTION

It was a long development since first light-emitting diode (LED). First red LEDs were applicable only as signaling due to their low output optical power. By successive development of technology and new material systems, LEDs with shorter wavelengths have reached the market. Lastly, the growth technology for blue, violet, and ultraviolet (UV) LEDs based on GaN has been mastered. Crucial moment for this was in the beginning of 90's when p-type doping for GaN has been developed. This allows production not only of blue LEDs but also white LEDs using combination of blue, red, and green LEDs or only blue LED together with material converting part of the radiation toward longer wavelengths which gives white light. White light has become to be interesting for lighting application but problem was insufficient optical power for such purposes. However, manufacturers have boosted LEDs output power and now so called Solid State Lighting (SSL) technology based on white light LEDs is booming. Forecasts indicate that progress in this segment will be even faster and most of traditional luminaires will be replaced by SSL luminaires. Improve in output optical power is achieved by increasing LED efficiency (or efficacy for white LEDs), which still hasn't reached its limit although currently the most efficient LED prototype is breaking the boundary of 300 lm/W.

Currently, the most investigated approach for improving LED extraction efficiency is using so called Photonic Crystal (PhC). In conventional semiconductor light emitting diodes (LEDs), the majority of generated light is trapped in high refractive index confinement layers due to the total internal reflection at the semiconductor/air interface. It was confirmed that photonic crystal (PhC) structures are very useful in the surface of different semiconductors [24] for fabrication of semiconductor based PhC devices as LEDs with light emission enhancement [25] and photodetectors [26]. Many groups reported the usage of PhC patterning for light extraction enhancement (LEE) of LEDs. The enhancement of the extraction efficiency in LEDs through the use of PhC requires a structure design that optimizes the interaction of the guided modes with the PhCs. For maximum extraction efficiency, there must be a strong coupling between the trapped waveguide modes and PhC structure [25, 27]. The optimization parameters of PhC are related to the vertical structure of the LED, such as confinement layers, position of the quantum wells and parameters of PhCs. The period of patterned PhC should be related to the photonic band gap. Generally, the PhC periods used for LEDs LEE are about several hundreds of nanometers. Firstly, the depth of etching plays very important role in cross-coupling of trapped modes with the leaky Bloch modes associated with the PhC. Likewise the size of PhC holes, mostly known as fill factor, is related to the photonic band gap of PhC and determines the extraction efficiency. From the point of PhC shape and geometry, the wide variety of PhC and photonic quasi-crystals were investigated. Moreover, the choice of PhC geometry plays more important role in determining of far-field radiation pattern diagram of the LED. Photonic quasi-crystals with high level of symmetry produce less directional emission [27].

In this work, four high resolution techniques on LED surface for patterning of two-dimensional photonic crystal are experimentally verified. Most effective technique with sufficient resolution and large area patterning is interference lithography (IL). Another technique uses advantages of polymer material called polydimethylsiloxane (PDMS) which can be easily treated, PhC patterned, and manipulated. For more precise PhC patterning electron and ion beam techniques can be used as electron beam direct write lithography (EBDWL) and focused ion beam (FIB). However, both techniques are time consuming and expensive processes achieving the resolution of tens of nanometers. In this work, we summarize the using of all these four techniques for patterning the PhC in the GaAs based multiple quantum well (MQW) LED.

Optical properties were investigated by light-current (L-I) characteristics and local characterization of emitted light in the near-field region. Optical method called Near Surface Light Emission Images (NSLEI) is introduced and used for PhC LED characterization [23].

DISSERTATION THESIS

1. Acquire knowledge about properties of light-emitting diodes and techniques of improving their efficiencies.
2. Study current state of the light-emitting diode characterization.
3. Analysis of the methods and standards related to measurement of electrical and optical properties of the light-emitting diodes.
4. Analysis of the electrical and optical properties of selected commercially available high-power white light-emitting diodes.
5. Analysis of the effect of the photonic crystal parameters on electrical and optical properties of the light-emitting diodes with photonic crystals.

Current State of PhC LED Characterization

Currently, effect of PhC on the LED properties is mostly studied on the modern LED structures based on GaN. This effect is also possible to investigate on different LED structures (e.g. those based on GaAs), but it is inevitable to take into account the properties of the materials where PhC is implemented, mainly the refractive index. PhC designs shown in Fig. 1 are the most commonly used for improvement of LED optical properties.

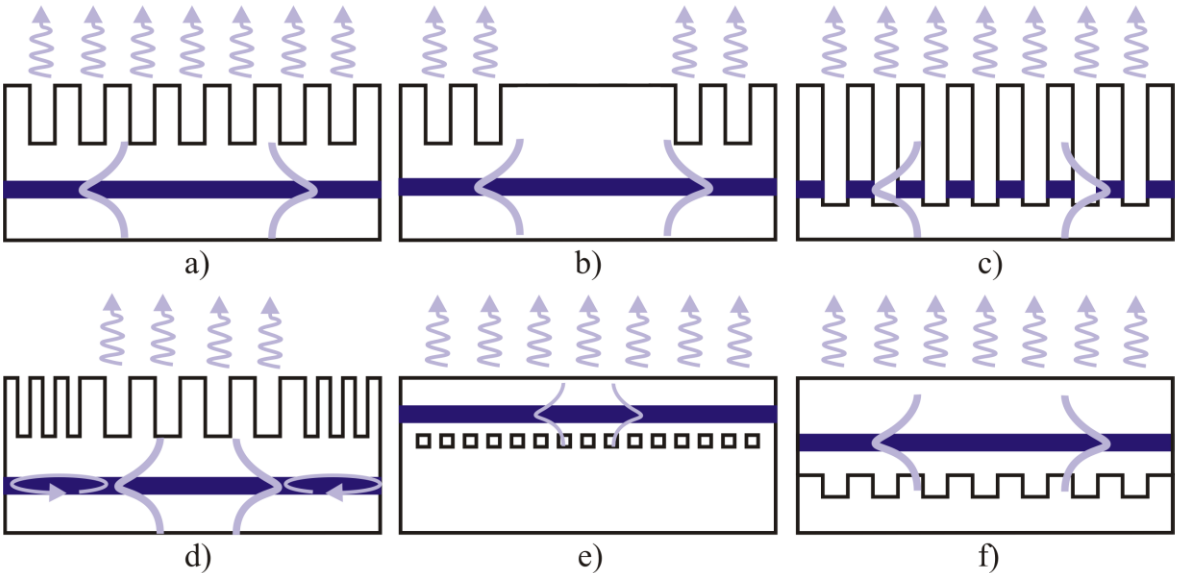


Fig. 1 The most commonly used PhC designs for improvement of electrical and optical properties of LEDs: a) uniform PhC, b) PhC LED with injection region, c) PhC etched through whole active region, d) PhC with distributed feedback, e) embedded PhC, f) PhC patterned substrate

Period of the structure shown in Fig. 1 a) is designed to act PhC as coupling grating for modes propagated in the active region of LED structure which acts as a planar waveguide as refractive index of active region is higher than refractive indices of boundary layers. This kind of PhC is usually two-dimensional (2D) with rectangular or triangular symmetry.

Effect of the rectangular and triangular symmetry as well as shape of etched holes on the electrical and optical properties of the LED is studied in [2]. Photonic crystals with different holes sizes and different shaped air holes including triangles, squares, and circles were fabricated on the top of GaN-based LED using EBL. PhC period was 400 nm. Cathodoluminescence (CL) of fabricated samples was studied. First experiment was aimed to comparison of the different shapes of the air holes and their effect on CL. As a reference, LED without PhC was measured. Results showed increase of CL peak intensities up to 43.2%, 105.9%, and 218.4% for samples with triangle, circle, and square holes PhC respectively. Next experiment investigated effect of periodical geometry on CL. CL intensities of square and hexagonal lattice PhC of the 200 nm square holes were compared. Results showed increase of CL spectra peak of 218.4% for square and 280.6% for hexagonal PhC. Next experiment was focused on the effect of etching depth on relative CL. Square and hexagonal PhC with square holes were fabricated with different depths.

Results showed that relative CL intensity is increases with increasing etching depth. Moreover, relative CL intensity for PhCs with the same etching depth is higher for PhC with hexagonal symmetry. Last experiment compared relative CL intensity of PhC with different sized holes. Size of investigated PhC holes was 100 nm, 150 nm, 200 nm, and 250 nm. Relative CL increases with increasing hole size up to 368% for 250 nm holes. Experiments proved that the best extraction enhancement of PhC LED can be achieved by PhC with hexagonal symmetry and square shaped holes. Size of these holes should be as large as possible as well as their etching depth.

Light extraction enhancement can be achieved in GaN based MQW LED structure also using relatively large PhC period [9]. PhC with square symmetry, 1200 nm period and 500 nm etching depth was chosen to extract high-order guided modes which can be extracted more efficiently than low-order modes. However, high-order modes can be extracted efficiently by large PhC periods with high etching depths. Radius of patterned holes was 360 nm. Light-Current measurements showed increase of light power output of PhC LED by 76% in comparison with non-PhC LED. Also external quantum efficiency increased from 18% to 32%.

FIB patterning of PhC was performed on GaN based MQW LED structure in [10].The photonic crystals of triangular air hole arrays were fabricated on the p-doped GaN layer. Total light emission area was 170x100 μm with FIB nano-patterned area size of 60x60 μm, which is 21% of the total light emission area. PhC period varied between 200 and 800 nm. Air holes diameter was 150 nm with etching depth of 50 nm and 200 nm. Micro photoluminescence (PL) measurements showed better results for samples with etching depth of 200 nm. The peak of the micro PL intensity spectra increased of 58% in comparison with normal LED. The integrated area under the illumination peak was increased by 75%. An additional set of LEDs was prepared with total light emission area of 65x65 μm with unchanged FIB nano-patterned area. This means that 85% of light emission area is covered by PhC pattern. Micro PL spectral measurements showed increase of peak emission by 91% and increase of integrated light emission of 106%.

Next approach, which can be used for LED light extraction enhancement with uniform PhC is deposition of ITO layer on the top of LED structure with consecutive texturing or PhC patterning [11, 12, 13, 14, 15].

GaN based MQW LED with ITO photonic crystal slab was studied in [11]. The refractive indices of GaN and ITO are close to each other so light emission can be coupled from GaN layer into ITO slab. To achieve easier manufacturability, PhC period in micron scale was chosen. The photonic crystal period was 3 μm with circular holes with diameter of 1 μm. Etching depth of PhC was 300 nm, which is equal to ITO layer thickness. Angular resolved spectral measurements of PhC LED were performed in Γ-K direction. Angular measurements show that PhC LED has a larger light emission angle (85°) than LED without PhC. Additionally emission peak appeared at 75°. Integrated light emission power was increased by 38% for PhC LED.

For PhC fabrication polystyrene (PS) microspheres were employed in [12]. PS microspheres colloidal monolayer was deposited on glass substrate. Then, the floating colloidal monolayer was picked up with an epitaxial wafer with 260 nm ITO film on it, followed by drying at 80 ◦C for 1 h in an oven. The wafer with PS spheres and ITO solution was dried and calcinated at atmosphere. PhC holes were hexagonally arranged with an opening diameter of about 1 μm and the maximum depth of 500 nm. As a reference conventional LED structure without PhC was fabricated. For investigation of light extraction enhancement light-current characteristics were measured. LED luminance intensity at 80 mA was enhanced from 118 mcd for conventional LED to 177 mcd for PhC LED which is enhancement of 50%. Spectral characteristics showed that peak intensity of PhC LED is 1.7 times higher than peak intensity of conventional LED.

In [13], two different wet-etching processes were used. First, 500 nm thick deposited ITO layer was etched in buffer-oxide etch (BOE) solution followed by annealing at 600°C for 2 min in air ambient. This is referred as "post-annealed ITO". Second, order of these processes is changed and this is referred as "post-etched ITO". AFM images shows that RMS roughness of post-etched ITO layer is 5.2 nm which is used as a reference to roughened post-annealed ITO layer with RMS roughness of 81.6 nm. This layer contains randomly distributed sphere-shaped protrusions with diameter between 250 nm and 1100 nm. Samples with three different etching times (7 s, 10 s, 12 s) were prepared for this kind of structure. Light-current measurements show that post-annealed ITO layer with all three etching times achieved increase in light output power in comparison with post-etched ITO layer. The highest increase in light output power (31.7% at 20 mA of forward current) was achieved at sample with etching time of 7 s, while this increase degrades with increasing etching time. Increase of 29.1% for 10 s etching time and 19.2% for 12 s etching time was achieved.

Approach with combination of ITO/ZnO layers prepared on the top of LED structure can also increase light output form LED as studied in [14]. One and two interface of textured ITO/ZnO layers was fabricated on the top of the LED structure. Fabricated one interface layers 200 nm thick ITO layer was evaporated and annealed at first. Then, a 100 nm thick ITO layer was evaporated for nanosphere formation. Finally, the methanol-dispersed ZnO nanoparticle was spin-coated onto ITO textured surface. Fabricated two interface layers 100 nm thick ITO nanoparticle layer was evaporated at first. Then 200 nm thick ITO layer was evaporated on the nanoparticle layer. Finally, ZnO nanoparticle was spin-coated onto ITO textured surface as in the previous case. Third sample was fabricated with only ZnO nanoparticle film deposited on the planar ITO film. L-I characteristics of all samples were measured. One interface of ITO/ZnO layer (Sample 1) showed increase in output optical power of 22.29% at 20 mA forward current. Additional enhancement of light extraction efficiency was achieved using two interface of ITO/ZnO layer (Sample 2). Improvement was of 35.54%. This was assigned to multiple light scattering at two different interfaces. Sample 3 with textured ZnO layer on the planar ITO film was fabricated to examine the effect of ZnO layer texturing on the p-GaN interface. Light extraction enhancement was of 20.89%, which is similar to one interface ITO/ZnO layer. This shows importance of two interfaces with textured layers in light extraction enhancement. All samples were compared to conventional LED without any interfaces on the top.

Similar approach was used also in [15] where polystyrene nanospheres with diameter of 500 nm were spin coated on the top ITO layer of LED forming hexagonal PhC. The diameter of the nanospheres was then reduced to 120 nm, 200 nm, and 280 nm. Subsequently, 100-nm thick ZnO layer was deposited on the ITO layer. Finally, nanospheres were removed by immersing in a CH_2Cl_2 solution. As a reference, LED with planar ZnO layer was fabricated. L-I characteristics of these four samples were measured. Results showed increase of light extraction improvement with increasing nanosphere diameter. This means improvement of luminance intensity of 7.9% for 120 nm, 12.5% for 200 nm, and 17.3% for 280 nm for 20 mA forward current.

Light extraction principle depicted in Fig. 1 b) is based on injection of the free charge carriers in the region without PhC. Then, generated guided modes are coupled from the active region by PhC. Advantage of such a structure in comparison with structure in Fig. 1 a) is more efficient free charge carriers injection into the active region as structure in Fig. 1 a) is uniformly covered by PhC and thus ohmic contact isn't ideal which leads to increase of its series resistance. Issue of light coupling from structures shown in Fig. 1 a) and b) is studied in [3]. Simulations show effect of PhC parameters on LED optical properties. Study doesn't take into account only PhC parameters but also parameters of LED structure. Two types of LED structures are compared here. First is GaAs based which active layer is generally single mode waveguide. Results for this LED structure show that the higher is the etching depth the higher is also extraction efficiency. Second type was GaN based LED structure. Layers of such a structure create planar waveguide with core of higher thickness which leads to more guided modes and thus to multimode planar waveguide. Results show that for this kind of structures there is need for higher etching depth than for GaAs based LED structures, which is technologically more difficult. Comprehensive study of light coupling from GaAs based LED heterostructures is performed in [4].

Although in approach for light extraction improvement shown in Fig. 1 c) PhC is etched through the whole active layer which partially disrupts this layer, there have been reported an improvement in light extraction efficiency in [16]. Studied LEDs were based on GaN with InGaN/GaN MQW. 220 nm thick transparent ITO film was deposited on the top of LEDs. PhC had square symmetry with 460 nm period. Effect of three different PhC depths was studied. First PhC was etched to the top ITO layer with etching depth 120 nm. Second PhC was deeper, reaching also top GaN layer with etching depth of 260 nm. Last PhC was etched through whole MQW with etching depth of 500 nm. The largest light extraction enhancement was denoted with 500 nm deep PhC. In this case the light output power was increased by 37% at 20 mA of forward current in comparison with reference LED without PhC. External quantum efficiency of LED with 500 nm deep PhC reached 11.6% while reference LED without PhC was evaluated as 8.5%.

Next possible approach how to improve LED light extraction efficiency using PhC is shown in Fig. 1 d). Whole LED emission area is covered by PhC. PhC is designed to couple modes guided in the waveguide. However, on the emission area boundary there is PhC with parameters designed to act as distributed feedback (DFB PhC). Light generated in the active layer and guided as a mode in the waveguide needs to pass certain distance (extraction length) to couple from the active layer. Light, which doesn't pass sufficient distance to couple from the active layer and reach PhC with distributed feedback properties, is reflected back by this PhC and can be additionally coupled from the active layer. PhC DFB has required effect only for light which is generated in distance from DFB PhC shorter than extraction length. Similar structure was studied also in [5]. The only difference in comparison with structure in Fig. 1 d) is that studied structure has DFB PhC etched through whole LED active layer. However, this isn't an issue, as active layer under DFB PhC is not used for light generation. Using this structure, light extraction efficiency was increased by 33.9 % in comparison with plain structure without PhC.

Structure in Fig. 1 e) seems to be another promising approach, because it has the best light extraction efficiency. It's LED structure with embedded PhC, which is prepared under the active layer of LED structure. Preparation of such PhC on the top of GaN based MQW LED structure is studied in [6]. This type of PhC is suitable especially for GaN based LEDs, where multiple waveguide modes are guided in the structure, because embedded PhC has higher extraction efficiency for higher guided modes than for fundamental mode. On the other hand, PhC in Fig. 1 a) is more suitable for LED structures based on GaAs, where only single (fundamental) mode is guided in the active layer. However, investigations in [6] show, that in the case where active layer above embedded PhC is thin enough, only single mode is guided in this region and can be effectively coupled also by embedded PhC. Study in [7] compares the extraction lengths of the surface and embedded PhC. Results confirms better higher extraction efficiency for embedded PhC and higher extraction length for surface PhC (55-120 μm) than for embedded PhC (64-80 μm).

Last commonly used approach for LED light extraction improvement is shown in Fig. 1 f). It is LED structure with PhC patterned on the LED substrate. Whole active LED structure is grown on this substrate. Such a structure prepared on sapphire substrate was studied in [17], where it's called photonic crystal-patterned sapphire substrate (PCPSS). Photonic crystal had triangular symmetry with period of 500 nm. Then, GaN based LED structure with InGaN/GaN MQW was grown on this substrate. As a reference, the same GaN based LED structure was grown on conventional sapphire substrate (CSS). Current-voltage measurements showed decrease of forward voltage in the case of LED with PCPSS in comparison with CSS LED. The forward voltage was measured at 20 mA of the forward current and its value was 3.17 V and 3.27 V for PCPSS LED and CSS LED respectively. Light-current characteristics showed 63% increase of the luminance intensity of PCPSS LED in comparison with CSS LED.

Similar study in [18] with PhC between the sapphire substrate and GaN epilayer of GaN-based near-UV MQW LED showed light emission enhancement of substrate patterned LED by a factor of 1.42 at 20 mA forward current and 1.25 at 100 mA of forward current Patterned PhC was 2D PhC with period of 600 nm. As a reference LED a sample without PhC was prepared.

Interesting study in [19] combines more of previously mentioned approaches. ZnO nano-structures were formed on ITO layer patterned on GaN based LED with non-patterned and patterned sapphire substrate (PSS). ZnO nano-structured layer was prepared using imprint process. As an imprint stamp soft PDMS stamp replicated from the Si master template. ZnO nanostructure has triangular symmetry with diameter of 250 nm and 700 nm period. Electroluminescence of non-PSS and PSS nanostructured LED was measured in vertical and 40° tilted direction at 20 mA forward current. Peak value increase in vertical electroluminescence was 20.5% and 19.0% for non-PSS LED and PSS LED respectively. For 40° tilted direction it was 41.3% and 56.0% for non-PSS LED and PSS LED respectively.

Design of the structures, which improve light extraction efficiency of LEDs is also verified by simulations in [28, 8, 29], where suitable PhC parameters as well as parameters of LED structure can be determined. In [28, 8], authors deal with additional approaches for light extraction efficiency improvement, which can be complementary to light extraction efficiency improvement by PhC. This applies for LEDs based on GaN with surface PhC. Near to LED active region $Al_xGa_{1-x}N$ boundary layer is grown. This layer has low refractive index, which leads to stronger light coupling in LED active region acting as a planar waveguide. Higher refractive index contrast can also cause excitation of more waveguide modes, which can be subsequently coupled by PhC from the active region to the air.

ACHIEVED RESULTS

1 PHC LED PREPARED BY INTERFERENCE LITHOGRAPHY

All samples investigated in this paragraph were prepared in collaboration with University of Žilina. Morphological properties of prepared PhC LED samples were studied by AFM. 3D image and 2D profile of investigated sample are shown in Fig. 2. AFM analysis showed good uniformity of patterned PhC across whole sample. Analysis of height profile perpendicular to refractive index change showed depth of etched holes as 104 nm with period of 600 nm. As this technology allows patterning only on relatively large area, metallization of LED covers also PhC patterning. This leads to larger metallization area and thus lower resistance of ohmic contact.

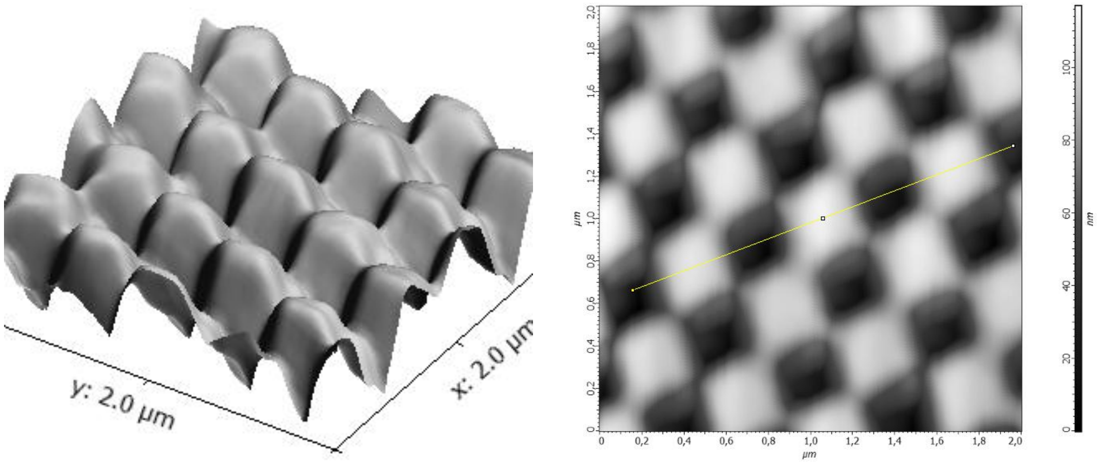


Fig. 2 3D image of investigated PhC LED (left) and 2D measurement with height profile (right).

Electrical and optical properties were measured by I-V and L-I characteristics. The measured I-V characteristics and L-I characteristics using integrated sphere are shown in Fig. 3 left and right respectively. Output optical power increased dramatically using PhC patterning in the LED surface with LEE of 265% in comparison with non-PhC LED. Origin of this improvement can be divided into two significant effects. First is surface roughness of the patterned LED surface. Using PhC patterning, roughness is higher and thus more light can be refracted in the air because conditions for total internal reflection on the air-AlGaAs interface were changed. The second effect, which plays an important role is, that active layer of the LED structure consisted of three quantum wells acts like a core of planar waveguide with coupled modes generated by recombination in active region. These coupled modes can be extracted into the air using a periodic grating etched to the top layer of the waveguide using photonic band gap effect. Etching depth of the PhC is important because evanescent wave, which can be extracted, has limited penetration depth and the higher etching depth the higher amount of light can be extracted. I-V measurement (Fig. 4 left) shows improved series resistance of PhC LED in comparison with non-PhC LED which is caused by metallization covering PhC and thus lower resistance of ohmic contact. [20].

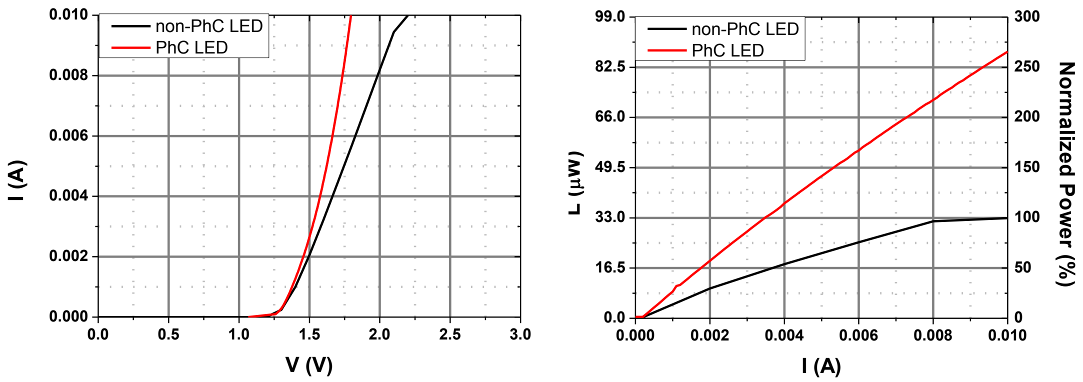


Fig. 5 I-V (left) and L-I (right) characteristics of investigated PhC LED.

2 PhC LED Prepared by PDMS Membrane (PDMS PhC LED)

Set of AlGaAs/GaAs LED samples with 2D PhC patterned PDMS membranes was prepared using technique described above. Period of 2D PhC with square symmetry patterned on the top of PDMS membrane was 495 nm and its depth was 100 nm. For better manipulation, PDMS membrane was prepared with thickness of ~30μm. Quantification of LEE of PDMS PhC LED samples over reference plain LED samples was made by light-current measurements (Fig. 4). Related to this technique, there are two mechanisms of improving LEE. The first is due to the lower refractive index of PDMS than refractive index of GaAs cap layer of LED, which leads to lower refractive index contrast on the top LED boundary. The second is related to PhC which causes light extraction of mode guided in LED active layer [21]. Accordingly, in Fig. 4 three different samples were measured (plain LED, plain PDMS LED, PhC patterned PDMS LED). ΔL_{PDMS} is LEE of plain PDMS LED over plain LED. ΔL_{PhC} is additional LEE of PhC patterned PDMS LED over plain PDMS LED. Then, ΔL_{TOTAL} is LEE of PhC patterned PDMS LED over plain LED. Measurements show LEE of 17.1% for plain PDMS LED and additional 16.4% for PhC patterned PDMS LED with total LEE of 36.4%.

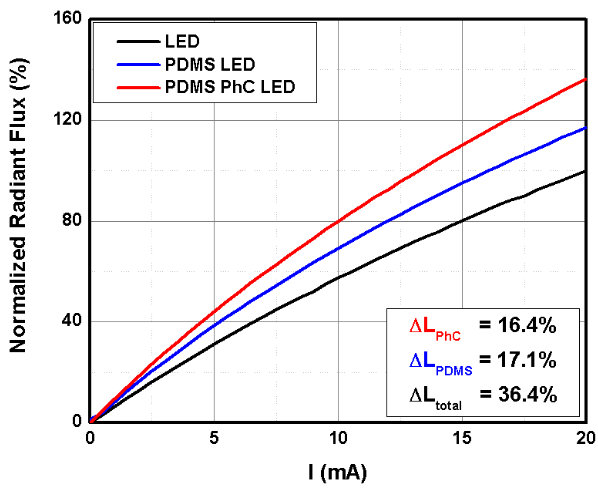


Fig. 4 L-I characteristics of plain LED, plain PDMS PhC and PhC patterned PDMS LED.

3 PhC LED Prepared by Focused Ion Beam

FIB enables high quality PhC structures. Circular shaped air holes are milled directly into the semiconductor by Ga ions and thus, resolution of the patterning can be as low as few nanometers. Depth of the air holes depends only on the ion dose impacting on the surface of the semiconductor. Advantage is that this method enables patterning of arbitrary shape with well-defined depth. It also doesn't need the etching process and pattern area can be very well defined. Disadvantage is limited patterning area and destructive character of the milling process.

Investigated PhC patterns were milled by FIB in collaboration with TU Ilmenau on the surface of AlGaAs/GaAs LED structure. Four different 2D photonic crystals from Fig. 5 were patterned. Fig. 5a,b show 2D PhC patterns with 750 nm period with square and triangular symmetries respectively. Patterns of 2D quasi-photonic crystals (QPhC) with Archimedean tiling A7 and A13 are shown in Fig. 5c,d. Advantage of this kind of structure is that it enables better decoupling of the light from the propagation plane in comparison with conventional 2D PhC with square symmetry. This is caused due to the properties of the crystal and it is also noticeable in the corresponding Fourier transforms. 2D PhC with square symmetry has four significant decoupling directions with corresponding vectors of the same size. Each of these vectors has its own representation in the direct lattice, which means that such PhC can decouple light in four directions with the same decoupling properties. On the other hand, QPhC with Archimedean tiling A7 and A13 enables up to 12 directions [1]. All PhC and QPhC were patterned with acceleration voltage of 30 kV and ion beam current of 20 pA. Patterns were milled in the area of 40 μm x 40 μm during 1800s. These conditions ensure the same ion dose for each sample. Non-patterned LED structure was used as a reference to LEDs patterned with PhC and QPhC structures. SEM images of four investigated LED patterns in Fig. 5 show that holes milled to the top of the LED structure were defined very precisely.

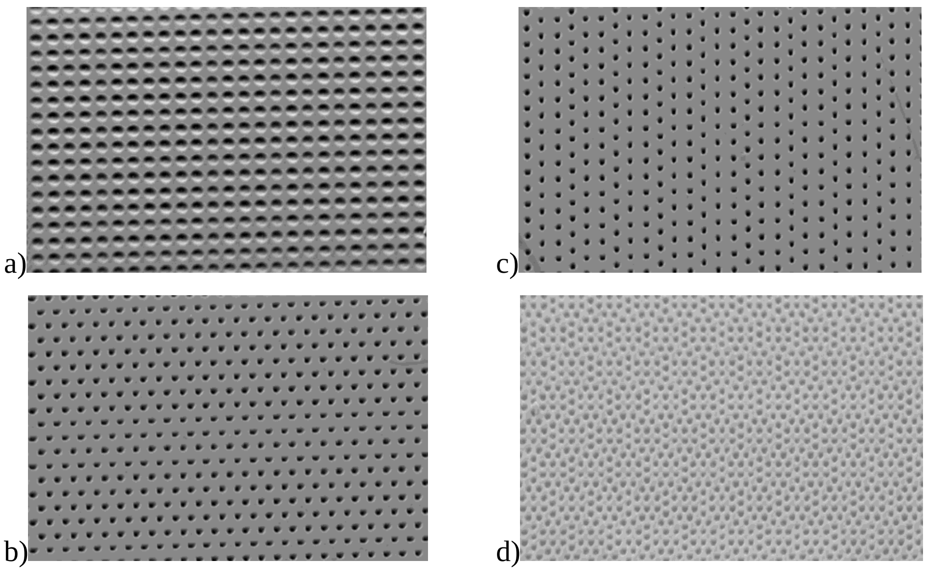


Fig. 5 SEM images of patterned structures prepared by FIB a) 2D PhC with square symmetry, b) 2D PhC with triangular symmetry, c) 2D QPhC with A7 symmetry, and d) 2D QPhC with A13 symmetry.

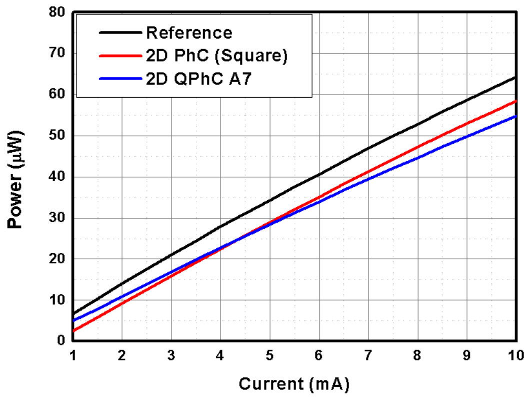


Fig. 6 Light-Current characteristics of investigated PhC and QPhC LED

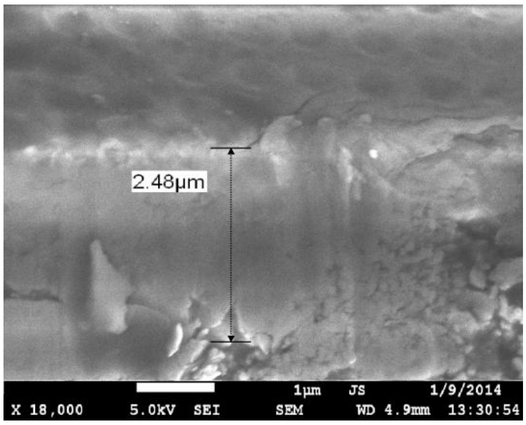


Fig. 7 SEM image on facet of PhC LED damaged by Ga ions during FIB patterning.

Fig. 6 shows L-I characteristics of LED with 2D PhC with square symmetry and LED with QPhC with Archimedean tiling A7. They show decrease of light output in the case of PhC and QPhC LEDs in comparison with reference LED. Light output decreased to 91% for PhC LED and 85.2% for QPhC LED for biasing current of 10 mA.

Impact of the ions on the LED sample led to its damage deep in the structure where Ga ions had been penetrated. This can be seen in Fig. 7 where damage is presented across the sample in the depth higher than 2.5 μm. We consider leakage current as another important factor which in combination with Ga ion damage can be more significant and leads to non-radiative recombination and decrease in light extraction efficiency for PhC LED compared to reference LED.

4 PhC LED Prepared by EBDWL

1D and 2D PhC symmetries were patterned by EBDWL on the top of LED in collaboration with Institute of Informatics SAS. Various optical properties of 1D and 2D PhC LEDs with different periods were studied. Additionally, properties of PhC LED were further enhanced by deposition of additional layer on the top of PhC.

4.1 1D Photonic Crystal

The MOCVD grown $Al_{0.295}Ga_{0.705}As/GaAs$ heterostructure of LED were spin-coated with a solution of hydrogen silsesquioxane (HSQ) electron beam resist (XR 1541-006) in methyl isobutyl ketone (MIBK). In order to get a high contrast and a good reproducibility, after the spinning the HSQ layer was prebaked on a hotplate for 2 min at 170 °C. The resulting thickness of the dry resist was 150 nm. The exposure was done using ZBA 23 (currently Vistec, Ltd.) e-beam pattern generator with 40 keV energy of electrons. We have chosen the standard and optimized exposure process where the resist shows the highest contrast: 500W / 5min substrate cleaning in O2 plasma; resist spin-coating at 1200 rpm.s-1; EBDW lithography; resist development at 21 °C for 120 sec in the AZ 326 MIF (Microchemicals) developer, and final rinse in DEMI H_2O for 10 sec. After this procedure a post-exposure bake at 350 °C in N_2 atmosphere took place. Optimal doses depend upon beam energy, desired resolution and film thickness. In order to eliminate possible organic residuals on the wafers a soft descumming was done in O_2 plasma at 1 Pa, 150 W, -180 V USB and 60 s.

1D PhC have been patterned by EBDW lithography and different periods of the gratings of 500, 600, 700, 800 and 900 nm were exposed. As variable energy e-beam lithography allows to control the electron penetration depth in HSQ, we carried out several tests with different exposure doses ranging from 140 to 320 μC/cm2 for all periods. Our aim was to obtain 1:1 line/space (L/S) ratio. The chosen samples were used for pattern transfer into the LED structures, and were subsequently etched in $SiCl_4$ plasma on the MICROSYS 350 equipment (45 W, -250 V, 4 min). The etch depth was 250 nm. The resist residuals after the etching were cleaned in diluted hydrofluoric acid.

For desired periods of 1D PhC the optimal dose was between 180-200 μC/cm2 giving the desired L/S ratio 1:1. For higher doses the L/S ratio was up to 5:1; for doses above 320 μC/cm2 the structures were completely overexposed. On the other hand, very low doses (up to 140 μC/cm2) led to insufficient resist change and during development it was partially diluted leading thus to resist thickness decrease (to up to one third of its initial thickness). Moreover, at low doses the pattern was deformed. In the doses interval between 140-180 μC/cm2 the line edge was rather sharp but the L/S ratio was still insufficient. Images from SEM and AFM in Fig. 8 show optimal patterned 1D PhC [22].

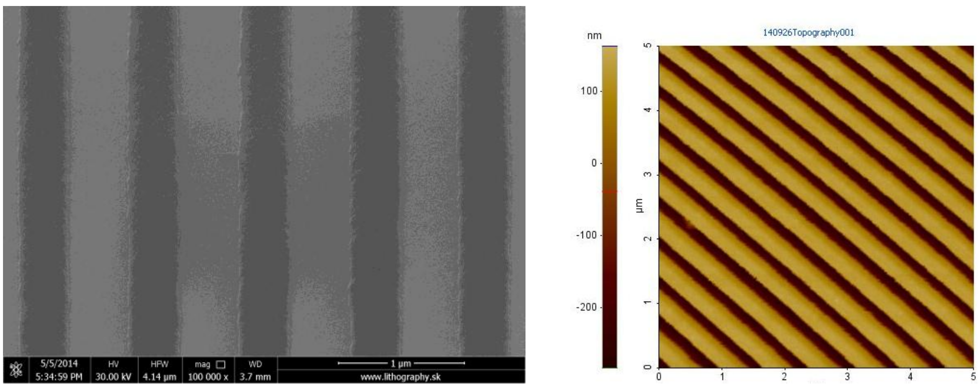


Fig. 8 SEM (left) and AFM (right) image of EBDWL patterned 1D PhC. [22]

1D PhC LEDs were studied by optical characterization measuring light-current characteristics by integrating sphere. LED samples with 5 different 1D PhC periods (500 nm, 600 nm, 700 nm, 800 nm, 900 nm) and 5 samples from each were measured. LEE was evaluated for each LED sample at 10 mA forward current in comparison with plain reference LED sample without PhC. All evaluated LEEs are summarized in Tab. 1. LEEs for particular PhC periods of studied LED samples were averaged. Light extraction enhancement was achieved for all studied samples for all patterned PhC periods. The largest LEE was achieved by PhC period of 600 nm with average LEE of 37.4 %. The smallest LEE was achieved by PhC period of 800 nm but still with average LEE of 21.7 %. Dependency of LEE on PhC period is shown in Fig. 9. Because coupling of guided modes by PhC from the LED structure must satisfy phase-matching condition this dependency is periodical. Therefore, this function was fitted using sine function with maximum of fitted function of 37.7 % at 582 nm and minimum of fitted function of 20.7 % at 842 nm. This gives half-period between minimum and maximum of 260 nm.

Tab. 1 LEEs at 10 mA forward current.

SAMPLE	**1D PhC PERIOD [nm]**				
	500	**600**	**700**	**800**	**900**
A	45.2	31.9	25.9	27.5	21.9
B	30.4	34.9	24.6	21.8	26.3
C	26.4	32.7	47.8	18.4	23.6
D	34.7	44.2	33.9	18.8	22.3
E	32.3	43.5	20.3	22.0	19.5
AVERAGE	33.8	37.4	30.5	21.7	22.7

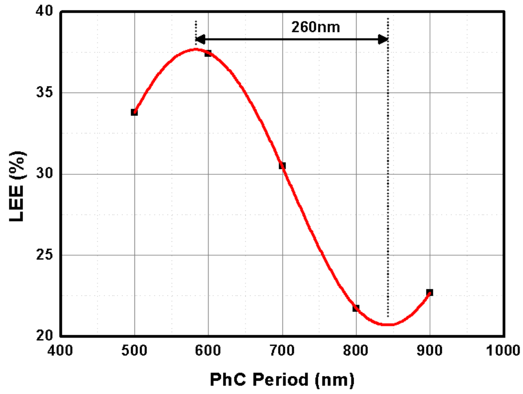


Fig. 6 LEE as a function of 1D PhC period.

In the next, light-current measurements performed by integrating sphere and by processing LED NSLEI were compared. Evaluated LEEs are summarized in Tab. 2. Higher LEE was achieved by method using integrating sphere for all studied 1D PhC periods. Together with the fact that NSLEI measures light emitted only from the top of the structure without side emission this implies that 1D PhC regardless of the studied PhC period improves LEE of PhC LED through the side emission. The highest difference in LEE between two methods is for 700 nm PhC period which means that PhC LED sample with this PhC period has the highest side emission, which contributes to LEE.

Fig. 10 shows 1D PhC LED NSLEI images captured at 10 mA forward current coded to colors. Violet color represents the lowest light intensity and it is increasing through blue, green, and yellow up to red. Boundary of the PhC area can be clearly seen as light generated in the active layer is extracted to the air when reaching the PhC boundary. This leads to increased light intensity at the boundary decreasing to the center of the PhC area due to decreasing coupling efficiency and lateral current distribution.

Tab. 2 Comparison of 1D PhC LEE measured by two different methods.

1D PhC PERIOD [nm]	500	600	700	800	900
LEE_{IS} [%]	33.7	44.1	47.7	22	22.3
LEE_{NSLEI} [%]	1.9	4.8	6.3	12.3	13.2
$\|DERIVATION\|_{MAX}$ [BIT/PX]	4.66	4.18	3.31	3.44	3.23

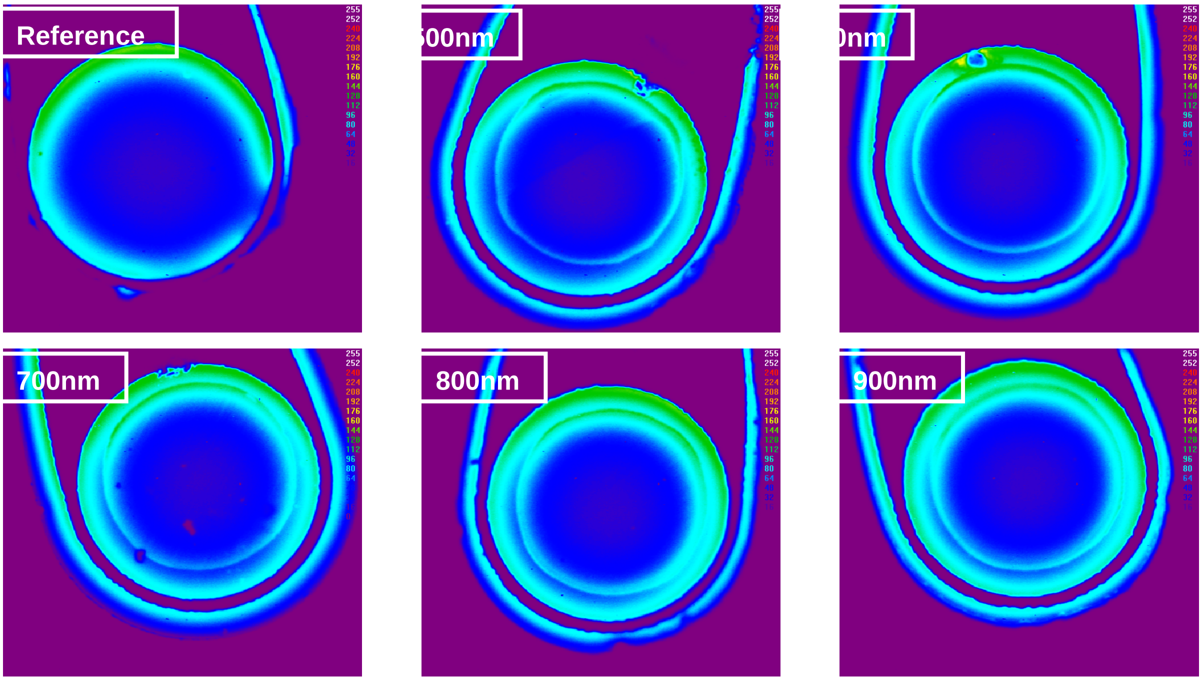


Fig. 10 NSLEI of reference LED and 1D PhC LEDs at forward current 10 mA.

Optical properties of PhC LED were further enhanced by deposition of additional layer on the top of PhC. Three different sets of PhC LED samples were processed. 1D PhC prepared by EBDWL was patterned on the top of LED samples in each set. Additionally, in one set a thin (semitransparent) Au layer was deposited on the top of patterned PhC and in the last set a thin ZnO layer was deposited instead of Au layer. Each set of samples was composed of 1D PhC with 3 different periods (500 nm, 600 nm, 800 nm) with 4 samples from each of them.

To confirm LEE of studied PhC LED samples the light-current characteristics were measured. Achieved results are shown in Fig. 11 for all sets of samples. LEE was improved for PhC LED with Au as well as ZnO deposited top layer for all studied PhC periods over bare PhC LED. The best overall results were achieved for PhC LED with Au layer with maximum LEE of 59%. The improvement of LEE of studied samples over bare PhC LED can be in general assigned to decreased ohmic contact resistance as well as improved LED lateral current distribution. Fig. 12 shows average LEE for investigated samples for given PhC periods. Evaluated results were interpolated by second order polynomial equation. For bare PhC LED as well as PhC LED with thin (<5nm) Au layer the best LEE was achieved for PhC period of 600 nm. For PhC LED with ~ 100nm thick ZnO layer the best LEE enhancement was achieved for PhC period of 500 nm. Interpolated dependency of LEE on PhC period for PhC LED samples with ZnO layer doesn't show any local maximum but a local minimum for LEE for the PhC period of 730 nm. This shows different behavior of 1D PhC LED samples for different top deposited layer [23].

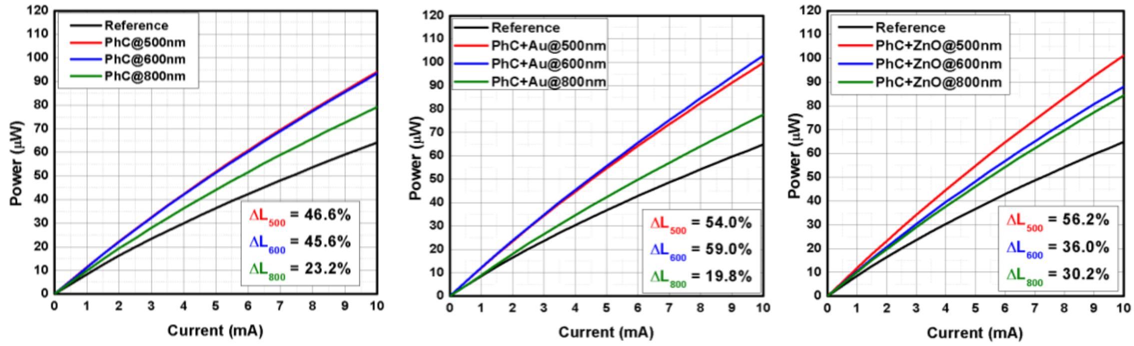


Fig. 11 LED with various PhC period prepared by EBDWL without conductive layer (left), covered by thin Au layer (middle), and covered by thin ZnO layer (right) [23].

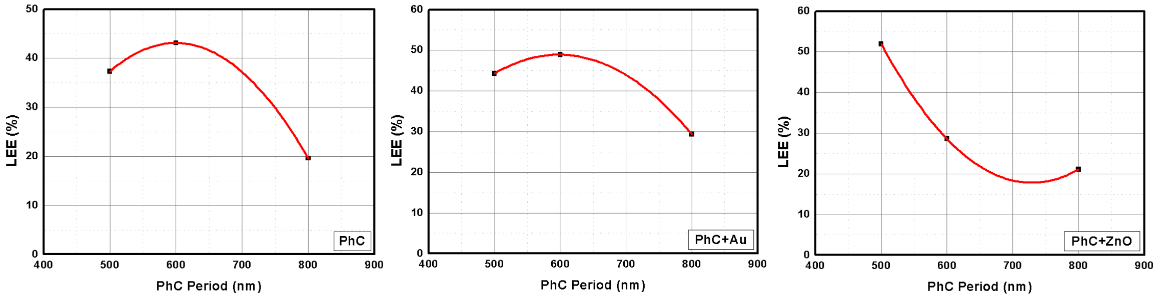


Fig. 12 Dependency of light extraction enhancement on PhC period for LED samples with different type of top layer. Bare PhC (left), PhC with Au layer (middle), and PhC with ZnO layer (right) [23].

4.2 2D Photonic Crystal

Patterning method used for the patterning 2D PhC was the same as described for patterning 1D PhC. Patterned 2D PhC was studied by AFM and SEM. These investigations (Fig. 13) confirm homogeneous distribution of 2D pillars with square symmetry with period of 500 nm. Height of the pillars was ~350 nm. 2D PhC LED samples were compared to the reference plain LED sample without PhC.

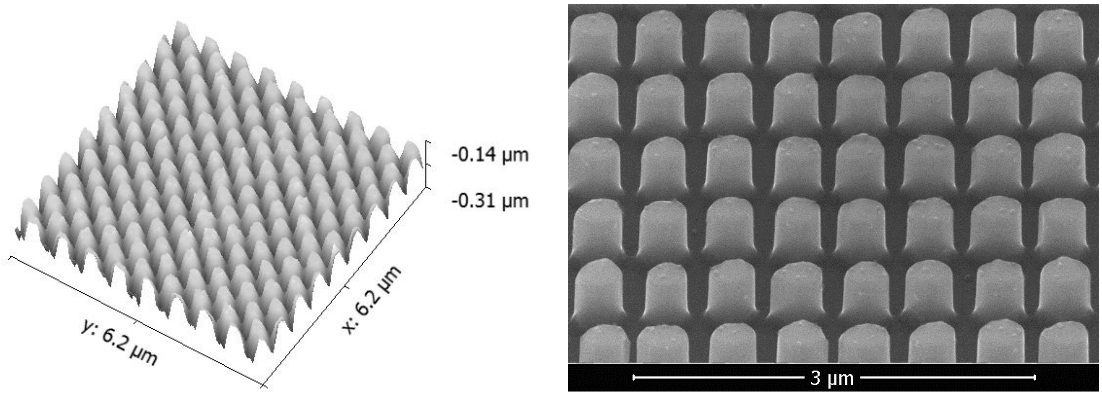


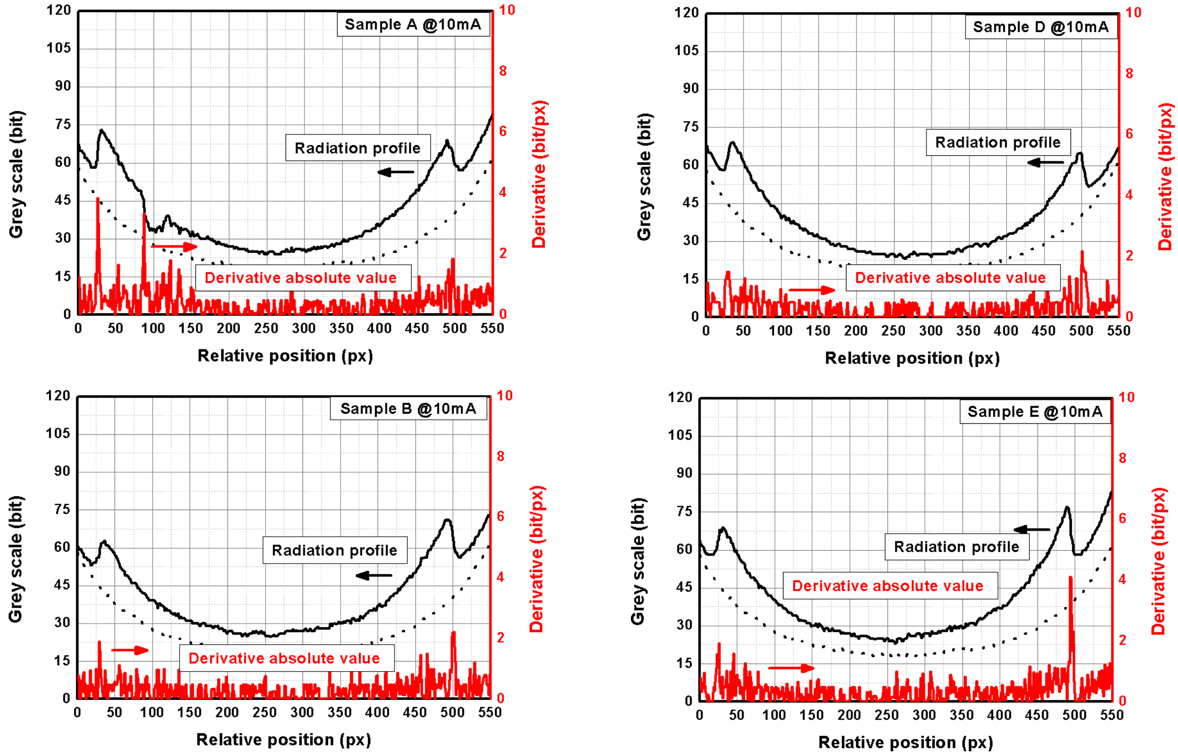
Fig. 13 AFM (left) and SEM (right) image of patterned 2D PhC.

Light-current characteristics were performed by integrating sphere and by processing 2D PhC LED NSLEI measurements for set of samples with period of 500 nm (A-E). The results are summarized in Tab. 3. Higher average LEE was achieved by NSLEI method (28.0 %) than by integrating sphere (20.8 %). As the NSLEI measures light emitted only from the top of the structure without side emission this means that 2D PhC LED with 500 nm PhC period with square symmetry improves LEE mainly by vertical emission.

Tab. 3 Comparison of 2D PhC LEE enhancement measured by two different methods.

Sample	A	B	C	D	E	**Average**
LEE_{IS} [%]	19.6	15.7	24.2	23.4	21.1	20.8
LEE_{NSLEI} [%]	21.1	25.6	34.7	29.2	29.4	28.0

Fig. 14 shows radiation profile from 2D PhC LED NSLEI images captured at 10 mA forward current. Light emitted near chip boundary is evident from increased emission by inner peaks. Boundary of PhC can be located by inner peaks in between first peak and last peak. Comparison of 2D PhC LED radiation profile (solid black line) and reference LED radiation profile (dotted black line) shows that the lowest LEE enhancement is presented in the middle of the radiation profile. This is consequence of lateral current distribution as well as related decreasing of light intensity from PhC boundary to the centre of the LED light emitting area [30, 31].



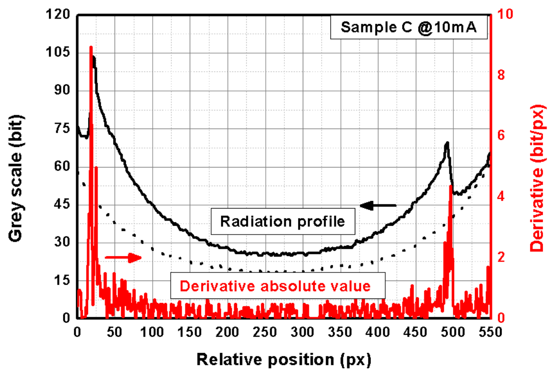


Fig. 14 Radiation profiles of 2D PhC LEDs with PhC period of 500 nm obtained from NSLEI measurements at 10 mA forward current (dotted line is reference sample).

SUMMARY

Four different methods for preparation of various types of PhC were described with their advantages and disadvantages. Interference Lithography (IL) is cost effective method which can be effectively used for patterning relatively large areas. Disadvantage of this method is that it can't be used for patterning arbitrary PhC shape and it can't be patterned on well defined areas. PDMS membrane patterning is another cost effective method with simple processing where PhC is patterned on the surface of thin PDMS membrane. Advantage of this method is possibility to pattern PhC on large area; disadvantage is limiting PDMS membrane thickness. Huge advantage of Focused Ion Beam (FIB) method is patterning arbitrary PhC on well defined area without need of photoresist. However, this area can be relatively small. Electron Beam Direct Write Lithography (EBDWL) is similar to FIB with its advantages and disadvantages, but in this method, PhC is patterned directly to photoresist.

Current state of research related to PhC LED and its characterization were summarized. In papers worldwide, many different approaches for PhC LED preparation have been used. For example uniform PhC, PhC LED with injection region, PhC etched through whole active region, PhC with distributed feedback, and PhC patterned substrate.

2D PhC was patterned by Interference Lithography on relatively large area. AFM analysis proved very good uniformity of the PhC across whole patterned area. L-I measurements showed improvement of LEE by the factor of more than 2.5. I-V measurements showed decrease of parasitic series resistance. This is caused due to metallization covering PhC which leads to higher ohmic contact area and thus to its lower resistance.

PDMS PhC LED samples were prepared using PDMS membrane with ~30μm thickness and 2D PhC patterning on the top. L-I measurements of three different sample types were compared. LED sample with non-PhC PDMS membrane showed LEE improvement of 17.1 % compared to bare LED sample. PhC PDMS LED sample showed additional 16.4 % of LEE improvement compared to non-PhC PDMS LED sample. This gives overall LEE improvement of 36.4 % for PhC PDMS LED compared to bare LED sample.

Four different 2D structure symmetries were patterned on the top of LED samples using FIB. PhCs with square and triangular symmetries were patterned. FIB is technique suitable not only for PhC patterning but also for QPhC patterning. Therefore, QPhCs with Archimedean tiling A7 and A13 were patterned. L-I measurements showed decrease of output optical power to 91 % for PhC LED and 85.2 % for QPhC LED. Analysis of SEM images of PhC LED facet showed damage of LED active layer by Ga ions penetrated deep into LED structure. Damage was presented across the structure in the depth higher than 2.5 μm. However, this technique showed that patterned structures can be defined very precisely with arbitrary symmetry. Therefore, together with the destructive character of this technique, it is more suitable for passive optical elements.

1D and 2D PhC symmetries were patterned to the top of the LEDs using EBDWL technique. 1D PhC with periods of 500 nm, 600 nm, 700 nm, 800 nm, 900 nm were patterned. LEE was evaluated for each LED sample and compared to plain reference LED sample without PhC. The largest LEE was achieved for PhC period of 600 nm (37.4 %) and the smallest LEE for PhC period of 800 nm (21.7 %). Dependency of LEE on PhC period was plotted and fitted by sine function with maximum of 37.7 % at 582 nm and minimum of 20.7 % at 842 nm. This gives half-period between minimum and maximum of 260 nm.

Light-current measurements were performed by integrating sphere and by NSLEI method. Better LEE was achieved by method using integrating sphere for all studied 1D PhC periods. The highest difference in LEE between two methods is for 700 nm PhC period which means that PhC LED sample with this PhC period has the highest side emission, which contributes to LEE.

Optical properties of 1D PhC LED were further enhanced by deposition of additional layer on the top of PhC (Au, ZnO). This technique was used to decrease ohmic contact resistance and improve LED lateral current distribution. The best overall results were achieved for PhC LED with Au layer with maximum LEE of 59 % for 1D PhC period of 600 nm. The highest LEE of 1D PhC LED with ZnO layer was 56.2% for 1D PhC period of 500 nm. Further, effect of deposited layer on dependency of LEE on 1D PhC period was proved. A local LEE maximum was observed for 1D PhC LED with Au layer at 600 nm 1D PhC period while a local LEE minimum was observed for 1D PhC LED with ZnO layer at 730 nm 1D PhC period, which shows different behavior of 1D PhC LED samples for different top deposited layer.

2D PhC with period of 500 nm was patterned on set of LED samples. Light-current characteristics were obtained by integrating sphere as well as by NSLEI method. Better average LEE was achieved by NSLEI method (28.0 %) than by integrating sphere (20.8 %), which means that 2D PhC LED with period of 500 nm and square symmetry improves LEE mainly by vertical emission.

Four different PhC patterning methods were evaluated in this thesis. From results, EBDWL method is considered as the most suitable method for LED PhC patterning as it offers the best compromise between LEE, PhC symmetry variability, patterned area size, and method invasiveness.

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