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Electrical investigations of hybrid OLED microcavity structures with novel encapsulation methods

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ABSTRACT

An electrical driven organic solid state laser is a very challenging goal which is so far well beyond reach. As a step towards realization, we monolithically implemented an Organic Light Emitting Diode (OLED) into a dielectric, high quality microcavity (MC) consisting of two Distributed Bragg Reflectors (DBR). In order to account for an optimal optical operation, the OLED structure has to be adapted. Furthermore, we aim to excite the device not only electrically but optically as well. Different OLED structures with an emission layer consisting of Alq₃:DCM (2 wt%) were investigated. The External Quantum Efficiencies (EQE) of this hybrid structures are in the range of 1-2 %, as expected for this material combination. Including metal layers into a MC is complicated and has a huge impact on the device performance. Using Transfer-Matrix-Algorithm (TMA) simulations, the best positions for the metal electrodes are determined. First, the electroluminescence (EL) of the adjusted OLED structure on top of a DBR is measured under nitrogen atmosphere. The modes showed quality factors of $Q = 60$. After the deposition of the top DBR, the EL is measured again and the quality factors increased up to $Q = 600$. Considering the two 25-nm-thick-silver contacts a Q -factor of 600 is very high. The realization of a suitable encapsulation method is important. Two approaches were successfully tested. The first method is based on the substitution of a DBR layer with a layer produced via Atomic Layer Deposition (ALD). The second method uses a 0.15-mm-thick cover glass glued on top of the DBR with a 0.23- μ m-thick single-component glue layer. Due to the working encapsulation, it is possible to investigate the sample under ambient conditions.

Keywords: Organic Light Emitting Diode, Distributed Bragg Reflector, Microcavity, Tamm State, Atomic Layer Deposition, Encapsulation, Alq₃:DCM

1. INTRODUCTION

The usage of organic materials in electronic devices increased after the demonstration of the first organic light emitting diodes (OLED).^{1,2} Organic materials have numerous advantages, e.g. broad absorption/emission spectra, low production costs due to thermal evaporation, the possibility to build flexible devices, and compact sizes. These advantages, especially the tunability³⁻⁵ due to the broad spectra, would be very interesting for a solid-state laser. Therefore, incorporating an organic material as active medium into a microcavity (MC) consisting of two Distributed-Bragg-Reflectors (DBR) seems to be a good choice. Also, MCs provide high quality factors (Q) which leads to lower laser thresholds.⁶

It was recently shown that achieving lasing in such devices with an optical pump mechanism is possible.^{7,8} For applications, however, it is not very convenient to require an additional pump laser. Therefore, an electrical pump process would be very desirable.^{9,10} The intensities needed to reach the lasing threshold in optical pumped samples suggests that the injected current densities need to be in the range of kA/cm².¹¹ Such high current densities rule out the usage of transparent contacts (e.g. ITO). Metal contacts can provide such current densities, but implementing those into MCs consisting of DBRs is difficult due to high absorption. Including metal layers into MCs has further impact on the behaviour of the device.¹²⁻¹⁴ A metal layer on top of a periodic dielectric structure, such as a DBR, forms a so-called optical Tamm state (TS) or optical Tamm plasmon which can be formed in s- and p-polarization. It is localized inside the DBR stop band with a parabolic dispersion, which lies within the light cone and can, therefore, be excited via photons.¹²

Optically pumped lasing has been achieved in spite of an included metal layer.¹⁵ Nevertheless, despite intensive

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studies^{16,17} an electrically driven organic solid state laser has not yet been realized.

In this paper we present the implementation of an OLED into a MC consisting of two DBRs. With the help of Transfer-Matrix-Algorithm (TMA) simulations, the optimal thickness and position of the metal and organic layers have been determined and optimized. Further, the experimental results, e.g. the OLED characteristics and the electroluminescence (EL) spectra, are presented. To realize measurements under ambient conditions, two new encapsulation methods are evaluated concerning their quality to encapsulate the diodes while not deteriorating the measurements.

2. SAMPLE FABRICATION AND EXPERIMENTAL SETUP

The investigated samples are build of two DBRs with an embedded OLED. The bottom DBR consists of 21 alternating $\text{TiO}_2/\text{SiO}_2$ -layers and each layer has an optical thickness of $\lambda_{\text{cav}}/4$. Here, λ_{cav} is the characteristic wavelength of the system. A reactive electron beam process is used to deposit the oxides on a pre-cleaned borosilicate glass substrate. This deposition takes place in a high-vacuum chamber with a base pressure of approximately $p = 10^{-6}$ mbar. To prevent the formation of unwanted suboxides, a partial oxygen pressure of $p_{\text{O}_2} = 2 \cdot 10^{-4}$ mbar is used. The corresponding refractive indices (at approx. 600 nm) of the materials are $n_{\text{TiO}_2} = 2.1$ and $n_{\text{SiO}_2} = 1.45$.

Fig. 1 depicts a microscope image of a DBR surface with a characteristic wavelength of $\lambda_{\text{cav}} = 610$ nm. It shows

Figure 1. Microscope image of a DBR surface. Holes and defects with varying size are randomly distributed.

a random distribution of defects with varying size. It is always possible to find a position without defect for optically pumped samples, but for electrically excited devices the excitation spot is fixed (where the two contacts cross). If such defects occur at the same spot as the contacts cross over, the diode has a short circuit. Therefore, the lateral extent of the diodes has to be decreased to increase the probability of working samples. Fig. 2 shows sketches of the adjusted metal shadow masks used for the bottom (a) and top (b) contact.

The OLED is deposited on top of the bottom DBR in an ultra-high-vacuum chamber system with a base pressure of 10^{-7} mbar. Both the organic materials and the metals are deposited via thermal vacuum evaporation.

At last, the sample is inserted into the previous high-vacuum chamber and the second DBR is evaporated with identical specifications. Due to different reasons (e.g. encapsulation, protection), some of the DBR layers may be substituted with other materials as will be explained in the following sections.

For the first encapsulation method a single-component-glue (EMCAST1500) is applied on a thin 0.15-mm-thick cover glass inside a nitrogen filled glovebox. The EMCAST1500 was chosen, because it has the same refractive index as the cover glass and thus avoids additional interfaces. The second encapsulation method is based on a

Figure 2. Metal shadow masks. Used to create the bottom (a) and top (b) contact of the OLED. The widths of the contacts are $200\text{ }\mu\text{m}$ / $100\text{ }\mu\text{m}$ to account for the defects of the DBR.

deposited layer using Atomic Layer Deposition (ALD).^{18–20}

The experiments to characterize the OLEDs are done in a nitrogen filled glovebox under room temperature since it is not possible to encapsulate the sample before the deposition of the top DBR. The EL spectra were recorded with a fibre optics setup and an Ocean Optics USB4000 spectrometer.

3. RESULTS

3.1 SIMULATION

To realize an electrically excited organic active medium embedded into a high quality MC, the idea was to start with a device structure which already shows lasing optically excited, and to adjust the structure to re-

Figure 3. a) Simulated transmittance spectrum for a MC with $\lambda/2$ organic layer (black dotted curve), $\lambda/2$ organic layer with two 25 nm thick silver layers (red dashed curve), and the adjusted (thickness, λ_{cav}) sample structure (green solid curve) b) Sketch of the adjusted device simulated in a) (not to scale).

Figure 4. Simulated electric field amplitude. The two white areas resemble the DBRs, the grey areas the contacts, and the green areas the OLED and protection layer.

alize an electrical pump process. Therefore, the adjusted structure consists of two DBRs with an embedded $\lambda/2$ Tris(8-hydroxyquinolino)aluminium (Alq₃) single-layer doped with 4-(Dicyanomethylene)-2-methyl-6-(4-dimethylaminostyryl)-4H-pyran (DCM). Here, the optimal doping ratio is 2 wt%.⁸ To electrically excite the device, two metal contacts are needed. Since it was possible to achieve optically pumped lasing with a 40-nm-thick silver layer in a similar sample structure,¹⁵ two 25 nm thick silver layers are used as contacts. To compensate for the huge red-shift (approximately 50 nm) and splitting of the cavity mode caused by metal, the characteristic wavelength λ_{cav} and the thickness of the cavity layer had to be adjusted as well. This was done based on TMA-simulations. Fig. 3 shows the simulated transmittance spectrum with $\lambda_{\text{cav}} = 604$ nm and a total thickness of the organic material of 155 nm. The TS1 of the adjusted device (solid green line) is located at 630 nm with a quality factor Q of approximately 850.

Fig. 3b) shows the complete structure of the device. Next, the single-layer Alq₃:DCM is transformed into a pin-OLED stack. Therefore, doped transport layers are necessary to improve the charge carrier injection and transportation. For the holes a 10-nm-thick N,N,N,N-tetrakis(4-Methoxy-phenyl)benzidine (MeO-TPD) layer is used which is doped with 2 wt% 2,2-(perfluoronaphthalene-2,6-diylidene)dimalononitrile (F6-TCNNQ) and for the electrons a 10-nm-thick Alq₃ layer is doped with caesium. In between the transport layers is a 125 nm Alq₃:DCM emission layer (EML) which is divided into two parts by a Tris(4-carbazoyl-9-ylphenyl)amine (TCTA) layer. The TCTA functions as electron blocker and ensures the recombination of the electrons and holes in the upper part of the EML. The lower part of the EML is electrically inactive but needed for optical control measurements and to provide a minimum of invertible DCM molecules.

The last adjustment is the substitution of the first top DBR layer (TiO₂) by an Alq₃:DCM layer with an optical thickness of $\lambda/4$. This substitution is possible because the refractive index of Alq₃:DCM is higher than the refractive index of SiO₂. The idea behind this additional organic layer is to protect the OLED from harmful radiation during the deposition of the top DBR.

Fig. 4 shows the simulated electric field amplitude for this device. The two grey areas depict the contacts and the two green areas portray the OLED (left) and the protection layer (right). The position of the TCTA layer in the current structure is based on this simulation. The recombination zone occupies only a small area of the EML and therefore, the TCTA shall ensure that the recombination zone and the maximum of the electric field coincide.

3.2 EXPERIMENTS

The samples are characterized after each production-step to gain better understanding of the complex device structure. In Fig. 5a), the IV characteristic of one representative diode on top of a DBR (red solid line) and in a MC (black dashed curve) can be seen. The characteristics are nearly identical, especially after the threshold voltage U_t is reached. In both cases U_t is approximately 15 V which is quite high compared to standard OLEDs where it is in the range of a few V. Nevertheless, considering the fact that it is a hybrid device and the thickness of the OLED has to be multiples of $\lambda_{\text{cav}}/2$, to fulfil Bragg's law, the increased onset voltage is justified. The External Quantum Efficiency (EQE) of the OLED is displayed in Fig. 5b). The maximum values of the

Figure 5. a) IV characteristics of the device without (red solid curve) and with (black dashed curve) top DBR. The IV characteristic after the deposition of the top DBR is nearly unchanged. An increased onset-voltage (approximately 15 V) was expected for this structure. b) EQE of three diodes (structure see Fig. 3b) with maximum values in the range of 1% to 2%.

EQE vary, dependent on the actual diode, but they are in the range of 1% to 2%. Compared to electrically optimized OLEDs with the same EML (Alq₃:DCM) achieving comparable EQEs,²¹ the values are surprisingly high.

The EL spectrum of an OLED is typically broad (~100 nm) and an EL spectrum of an OLED on top of a DBR

Figure 6. a) EL spectra of the device without top DBR. The TS1 is located at 643nm and the TS2 at 700nm. The corresponding Q -factors are $Q_{\text{TS1}} = 60$ and $Q_{\text{TS2}} = 60$. b) EL spectra of the device with top DBR. The TS1 shifts to 630nm and the TS2 to 668nm. The corresponding Q -factors increase to $Q_{\text{TS1}} = 600$ and $Q_{\text{TS2}} = 120$.

should behave similar, but as can be seen in Fig. 6a) there is not one broad emission peak from the DCM but two relatively sharp cavity modes. This is caused by the metal top contact which also functions as mirror forming a cavity with the bottom DBR/contact. The two modes are located at 643 nm (TS 1) and 700 nm (TS 2) and have quality factors of $Q_{TS1} = 60$ and $Q_{TS2} = 60$. Compared to the estimated Q -factors in the simulation (see Fig. 3), the Q -factors of both modes are very low. However, the top mirror in the simulation was a DBR and in this EL spectrum it is only a 25-nm-thick silver layer. Therefore, the Q -factor should increase after the deposition of the top DBR. This would prove the necessity of the top DBR despite its quite harmful deposition method, because the Q -factor gives a direct link to the laser threshold which means that with increasing Q -factors the laser threshold decreases, requiring lower pump intensities to reach it.

The EL spectrum of the same device with top DBR is shown in Fig. 6b). Two main changes can be seen: At first, the spectral position of the modes shifts. The TS1 is now located at 630 nm which fits the predicted spectral position of the simulation and the maximum of the emission spectrum of the DCM. The second obvious change is the increase in the Q -factors. The Q -factor of the TS1 increases by a factor of ten to $Q_{TS1} = 600$ and for the TS2 it doubles to $Q_{TS2} = 120$. Q -factors in the range of a few hundreds are a very good step considering the 50 nm of silver inside the MC. The simulation suggests that it is even possible to further increase the Q -factors but this goes probably hand in hand with improving the production steps of this complex device.

3.3 ENCAPSULATION METHODS

The next step to better characterize the sample is to find a suitable encapsulation method. In our case suitable means that the encapsulation has a high optical quality and is very thin in order to allow to get close to the surface e.g. with objectives. If it is possible to get close to the surface objectives with higher numerical apertures can be used which further allows measuring larger opening angles.

We tested two different methods and compared them. The first was inspired by the standard encapsulation

Figure 7. a) Sketch (not to scale) of the sample structure with ALD-encapsulation. b) Sketch (not to scale) of the sample structure with cover glass and glue encapsulation. c) Microscope image of a sample encapsulated with a cover glass.

method used in OLEDs: A glass substrate is glued on top of the device. Instead of the standard OLED encapsulation glasses we used a very thin (0.15 mm) cover glass. This is possible, because on top of the OLED is a DBR which functions also as a barrier between the OLED and the glue and prevents the glue from penetrating through it. As glue the single-component glue EMCAST1500 is used. The sample structure with encapsulation is shown in Fig. 7b).

The glue was spin-coated onto the cover glass and put on the sample. Afterwards, the glue was hardened using UV-radiation. At the edge of the cover glass an area was protected from the glue (see section 2 of Fig. 7c)

Figure 8. IV characteristics of a diode a) encapsulated with glue and cover glass and b) not encapsulated. The characteristics of the encapsulated diode remains constant whereas the onset voltage of the diode without encapsulation increases.

Figure 9. IV characteristics of a diode encapsulated using an ALD top DBR layer. Even after a total storage time over a month the characteristics do not change.

to build an air cavity above the device. The air cavity creates numerous Fabry-Pérot modes, while optically excited. This allows to extract the thickness of the final glue layer by evaluating the free spectral range. It turns out that the glue layer is only $0.23\ \mu\text{m}$ thick which is negligible compared to the thickness of the cover glass.

To prove the effectiveness of this method, the IV-characteristics of one diode from section 1 (encapsulated) and from section 3 (not encapsulated, see Fig. 7c) are shown in Fig. 8. The diodes are always measured simultaneously. The first measurement, before the encapsulation, was done in a nitrogen filled glovebox to protect the OLED. Here, both diodes (see Fig. 8 a and b) show identical characteristics. After the encapsulation, the sample was stored and measured under ambient conditions. For the encapsulated diode (Fig. 8 a), the characteristics remain constant. The diode without encapsulation on the other hand has an increased onset-voltage which proves the need of an encapsulation and shows that this method is working.

The second encapsulation method tested with this device structure is an integrated ALD-layer (see Fig. 7a).

Figure 10. a) Microscope image of the cross over from the bottom (horizontal) and top (vertical) contact. b) Microscope image of the same region as in a) but electrically excited. c) Horizontal intensity distribution. Homogeneous distribution and no waveguiding into the bottom contact c) Vertical intensity distribution. Homogeneous distribution with waveguiding into the top contact. The intensity at the interface between two and one metal has a sharp increase but declines after a few μm .

Due to the different growth of layers deposited via ALD compared to physical vapour deposition they can be used to encapsulate devices.²² Therefore, using an ALD-layer as one of the DBR layers would encapsulate the device without changing optical or electrical properties.

An Al_2O_3 -layer was deposited onto a silicon-wafer using plasma-ALD in order to determine the refractive index $n_{\text{Al}_2\text{O}_3}(\lambda)$ by means of ellipsometry to check the possibility of substituting it with the low-refractive-index-material of the DBR (SiO_2). This would allow to use it as the second top DBR-layer. The refractive index for the characteristic wavelength $\lambda_{\text{cav}} = 600$ is approximately 1.62 which is a bit larger than the refractive index of the SiO_2 -layer but lower than the refractive indices of the organic materials and TiO_2 . Therefore, it was possible to include it into the top DBR as low refractive index material.

Fig. 9 shows the IV-characteristics of a diode during its several production steps starting with an OLED on top of a DBR and ending with an OLED in a MC with an ALD layer as second top DBR layer. It is clearly visible that the characteristics are stable even after storage times of one month under ambient conditions.

So both encapsulation methods work for this kind of device and can be used to investigate the samples under ambient conditions.

The first experiment after the successful tests of the encapsulation methods was the investigation of the light emitting areas under a microscope. This test yields important information for future investigations. Fig. 10 shows a microscope image, where the contacts cross (a) and electrically excited (b). The contact-sizes are $200 \mu\text{m}$ for the bottom contact and $100 \mu\text{m}$ for the top contact. The graphs in Fig. 10 c) and d) depict the horizontal (c)

/ vertical (d) intensity distribution. Both clearly show a homogeneous distribution and not higher intensities at the edges, due to thinner metal stripes, which could be expected. There is wave-guiding into areas where only the top contact is (Fig. 10b) which explains the higher borders of the plateau. Due to the asymmetry of the sample structure (Alq_3 :DCM next to the top contact but not to the bottom contact) there is no wave-guiding into the bottom contact.

4. CONCLUSION

We have successfully implemented an electrically excitable organic LED into a MC. We reached Q -factors of $Q = 60$ for a MC consisting of a bottom DBR and a 25-nm-thick silver top mirror. After depositing the top DBR the Q -factor increased to $Q = 600$ (with two 25-nm-thick silver layers inside the MC) which proves the usefulness of the top DBR despite the harmful evaporation method. The EQE of the OLEDs is in the range of 1 % to 2 %. To enable investigations under ambient conditions two novel encapsulation methods with different advantages have been successfully tested. The first method, based on a cover glass and the single-component-glue EMCAST1500 is very time efficient and has a low risk of device damage. The second one, based on a ALD layer inside the top DBR, adds a further production step which increases the production time and the probability that the sample gets spoiled but it is very convenient to include the encapsulation into the top DBR. The device has a homogeneous intensity distribution where the contacts cross.

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