

New UV Technology for Point-of-Use Water Disinfection

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ABSTRACT

A new class of semiconductor-based Deep Ultraviolet Light Emitting Diodes (DUV LEDs) is emerging as a disruptive technology for point-of-use water disinfection. Sensor Electronic Technology, Inc. has pioneered compact DUV lamps based on III-Nitride semiconductors (GaN, AlN, InN and their alloys). These semiconductor-based DUV light sources offer narrow spectral power distribution (<12 nm FWHM) with peak emission wavelengths from 227 nm to 340 nm. CW optical output power of up to 100 mW (at 275-280 nm wavelengths) can be achieved using a single, hermetically sealed lamp with a footprint of about 1 square inch. We report on a record-breaking performance of an all-LED point-of-use water disinfection system that utilizes our DUV lamps with peak emission wavelengths in the range from 270 nm to 280 nm. We demonstrated reduction of *E. coli* bacteria concentration by 5.5 LOG for 0.5 liter/min flow and 3 LOG reduction for 2 liter/min flow.

Keywords: *UV disinfection, light emitting diodes, point-of-use water purification*

1 INTRODUCTION

Water is the basic and rapidly diminishing resource needed for life on the planet. Ultraviolet disinfection is an attractive alternative to traditional chemical processes, which require temperature and pH control and often create substantial amounts of disinfection by-products. Currently, low- and medium-pressure mercury vapor lamps are the only UV light sources used for water disinfection in municipal facilities and smaller scale industrial and residential air and water purification systems. Deep UV (DUV) semiconductor light emitting diodes (LEDs) represent new class of UV light sources for point of use (POU) water purification that are environmentally safe, easily reconfigurable, compact, robust, and cost effective. These mercury-free devices can be directly powered by low-voltage sources such as batteries or solar panels. Moreover, the spectral power distribution of multiple chip LED lamps can be controlled by appropriate electronics, enabling new applications in water, air and food disinfection as well as in biomedical diagnostics and treatment.

Sensor Electronic Technology, Inc. has developed DUV LEDs and multiple chip LED lamps based on various hermetically sealed packaging platforms. Single chip UVTOP[®] LED devices offer the flexibility of incorporating

small (0.35 inch in diameter) light source operating at low CW voltage (around 6 V) into a complex system. UV output from such devices can be coupled to the optical fiber enabling small spot excitation and light delivery. For applications requiring UV emission with higher optical power density, such as curing or disinfection, multiple chip DUV LED lamps with a footprint of about 1x1 inch have been developed. These DUV LED lamps emitting at 275 nm (+/-5 nm) exhibit CW optical power in excess of 50 mW with dissipated power around 10 W. In this paper we review current status of DUV LED light sources and discuss results of experiments carried out using DUV LEDs installed in disinfection and bioanalytical setups.

2 DEEP UV LED FABRICATION

2.1 LED Structure

The LED structures were grown on basal-plane sapphire substrates using a combination of metalorganic chemical vapor deposition (MOCVD) and proprietary and patented migration-enhanced MOCVD (MEMOCVD[®]) deposition processes.[¹] Trimethyl aluminum (TMA), trimethyl gallium (TMG), trimethyl indium (TMI), and ammonia (NH₃) were used as precursors with silane (SiH₄) and bis-cyclopentadienyl magnesium (Cp₂-Mg) used for n- and p-doping. All structures were grown using high-quality AlN buffers and AlN/AlGa_xN strain relief superlattices grown by MEMOCVD[®]. These layers were followed by a 3–4 μm-thick Si-doped n⁺-AlGa_xN, a multiple-quantum-well (MQW) active region, a Mg-doped p-AlGa_xN clad/blocking layer, and a patent pending graded p-AlInGa_xN p-contact layer. Al molar fraction in AlGa_xN MQW region and n- and p-emitter/clad layers was varied in order to obtain emission wavelength from between 230 nm and 365nm. The LED active regions used very thin (down to 1.5 nm) MQWs with the ground state energy above the energy range, where the bands are bent by the polarization fields. Such design substantially increased the oscillator strength due to better overlap of electron and hole wavefunctions and provided very efficient carrier confinement eliminating the need for a conventional electron blocking layer and reaching performance comparable to that expected from UV LEDs on non-polar substrates. In addition, the carrier capture into quantum wells was dramatically improved by using heterostructures designed for resonant coupling to polar optical phonon energy. Our photoluminescence studies confirmed that the 280 nm structures designed using this

patented phonon engineering approach provided very high (as high as 60%) internal quantum efficiencies.

Prior to processing the wafers were in-situ activated in growth chamber. Device fabrication included standard processing steps such as photolithography, dry etching and metal deposition. Chlorine-based plasma reactive ion etching was used for mesa formation. Electron beam evaporation was used for depositing Ti/Al/Ti/Au n-contact and Ni/Au p-contact metals. After deposition, thermal annealing in forming gas at 850–900 °C (depending on the Al composition of n-AlGaIn layer) and 500 °C was carried out for n- and p-contacts, respectively. Finally, dielectric isolation layer was deposited prior to chip separation.

2.2 UVTOP® devices

After processing the DUV LED chips were then diced and flip-chip packaged on SiC heatsinks to provide efficient heat dissipation and improved light extraction. For low power we developed UVTOP® product line, which is based on TO-39 headers capped with flat window or lens cup (see Figure 1) depending upon required angular distribution of UV light intensity. Currently, UVTOP® includes devices with the single emission wavelength from 245 nm to 365 nm as shown in Figure 1. UVTOP® devices operate at 20 mA CW with typical operating voltage ranging from 4.5 V to 7 V and output power from 0.1 mW to 2 mW depending upon the emission wavelength. For applications requiring UV light delivered to small excitation spot UVTOP® devices coupled to multimode UV optical fibers or SMA-type fiber connectors were developed.

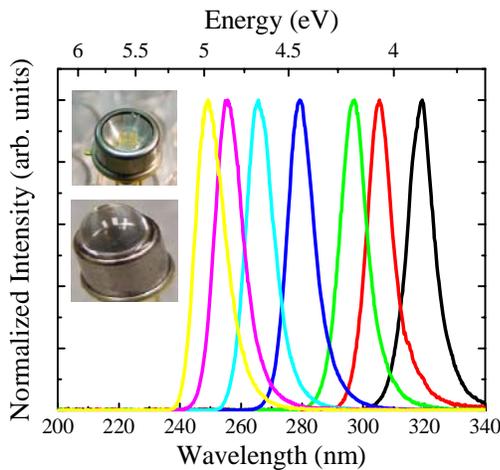


Figure 1. UVTOP® devices and their normalized spectral intensity distributions.

2.3 High power LED lamps

For high-power operation, arrays of up to 72 LED chips were assembled in the modified TO-3 packages with either internal thermo-electrical coolers or copper heat sinks. Temperature of the TO-3 package was monitored using a built-in thermistor. As seen from Figure 2, the CW output

power in excess of 80 mW was measured for these LED lamps at the pump current close to 950 mA. Inset to Figure 2 shows the spectral power distribution of the LED emission peaking at 279 nm. Enhanced heat dissipation using only passive cooling resulted in the case temperature overheating of only 12 °C at the pump current of about 1 A.

The output optical power exceeding 55 mW was demonstrated for lamps with peak emission wavelength in the range from 270 nm to 272 nm. These results represent the highest output power reported for sub-300 nm wavelength semiconductor light source.

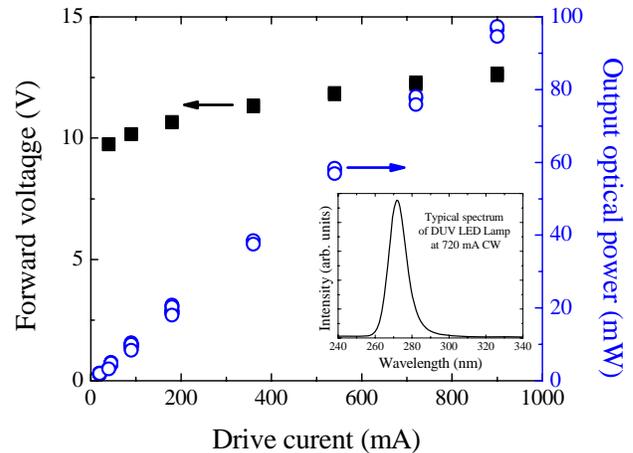


Figure 2. CW output power vs. current for 270-280 nm DUV LED lamps. Inset shows emission spectral distribution for the typical lamp at 720 mA

3 POINT-OF-USE UV WATER DISINFECTION

3.1. Dependence of germicidal effect on dose and wavelength

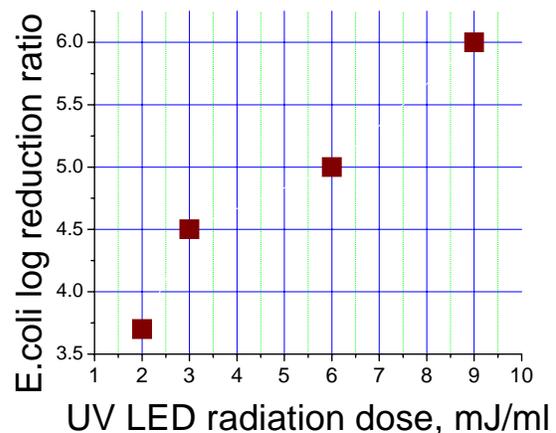


Figure 3. Germicidal effect of 280 nm UV LED light with 2,3,6,9 mJ/ml dose of irradiation

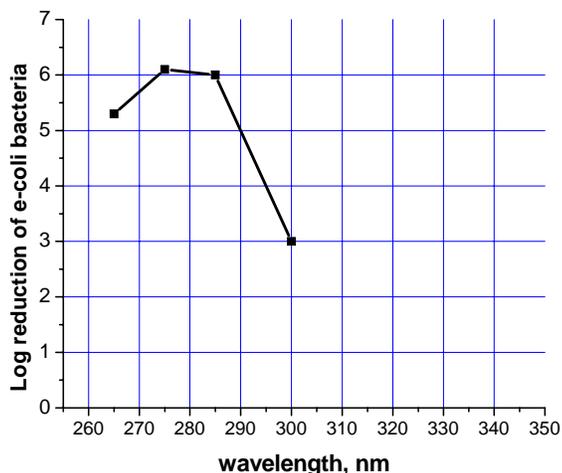


Figure 4. Germicidal effect vs. wavelength with similar UV radiation dose of 9 mJ/ml

Preliminary experiments (Fig.3 and 4) were made to estimate UV LED power levels and most effective spectral band needed to achieve required bacteria eradication effectiveness. Water disinfection system utilizes our DUV lamps with peak emission wavelengths in the range from 270 nm to 280 nm. The system consists of a 3 inch diameter and 6 inch long disinfection chamber equipped with 4 multi-chip DUV LED lamps mounted on one end of a cylindrical chamber. Each lamp was rated to deliver up to 60 mW output optical power at 500-700 mA driving current with forward DC voltage of 10-11 V (see Fig. 1). We performed water disinfection experiments using flowing contaminated tap water and DUV LED lamps with the total input optical power in the range from 34 mW to 68 mW inside the disinfection chamber.

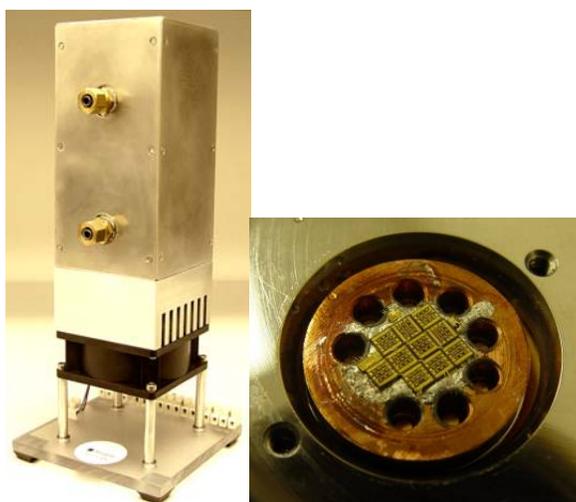


Figure 5. Water disinfection chamber and DUV LED lamps mounted to the bottom plated of the disinfection chamber.

3.2. Microbial characterization of DUV LED disinfection unit

Our research was focused on microbial characterization of disinfection unit with human and aquatic pathogens. In testing of UV disinfection apparatuses the standard objects are usually bacteria *Escherichia coli* and MS2 virus (bacteriophage).

Our initial germicidal testing was carried out with *E. coli* bacteria. *E. coli* (characteristic size of 0.5-2 μm) is a Gram negative rod-shaped bacterium from Enterobacteriaceae family, which includes well known pathogens as Salmonella and Shigella. In 1892 it was proposed to use *E. coli* as a common indicator because it is abundant in natural water and wastewater and has an ability to be easily detected.

E. coli (ATCC 11303) was obtained from American Type Culture Collection. Bacteria were grown in LB Broth™ to mid-log phase. Several aliquots were transferred onto agar plates and were incubated for 24 hours at 37 °C. After incubation an individual colony was inoculated into LB Broth for 24 hours at 37 °C and then used for preparation of dilutions with concentrations ranged from approximately 2.5×10^2 CFU/ml to 2.5×10^6 CFU/ml in a final volume of 1 L of deionized H₂O. The test bacterial suspension was mixed very vigorously with test organisms. Pure water samples and serial dilutions of control samples from each liter of water were collected for determination of *E. coli* initial concentration before UV irradiation.

Sample volume was contained in funnel, which was connected to the rotameter for the flow rate measurements,. Output of the rotameter was connected to the input port (bottom) of the prototype UV chamber (see Figure 5). Four different flow rates were used in disinfection experiments: 0.5 LPM (0.13 GPM), 1 LPM (0.26 GPM), 1.5 LPM (0.4 GPM), and 2 LPM (0.53 GPM). The output current and device voltage were monitored during each experiment. LED lamps were operating at 200 mA each.

After UV treatment samples were collected and several proper dilutions from each sample were used for determination of *E. coli* survival concentration after UV irradiation. All test suspensions were processed immediately after irradiation. For determination of *E. coli* concentration we used Colilert-18™ method (IDEXX Laboratories, Inc.) approved by US EPA.² After experiments appropriate aliquots of irradiated solutions were mixed with deionized water (final volume was 100 ml) and reagent was added. Then the solution was poured into Quanta-Tray/2000, sealed and incubated at 35 °C for 18-22 hours. After incubation the enumeration of bacteria as Colony Forming Units per ml (CFU/ml) was done.

Results of bacterial testing are presented in Figure 6. Logarithmic reduction of viable bacteria was calculated as $\text{LOG} (N_b/N_a)$, where N_b and N_a are the concentrations before and after irradiation, respectively. As seen from the Table 1, for each flow rate samples with different initial bacteria concentration were used. For some concentrations

the survival concentrations was not determined since amount of *E.coli* in the treated sample was below the lower limit of determination for Colilert-18 method.

SUMMARY

UV water disinfection was successfully demonstrated for various flow rates with the compact unit based on semiconductor DUV LED lamps. Prototype reactor was shown to be effective of both *E.coli* bacteria and MS2 bacteriophage UV disinfection at very moderate UV power levels and very low input electrical power.

REFERENCES

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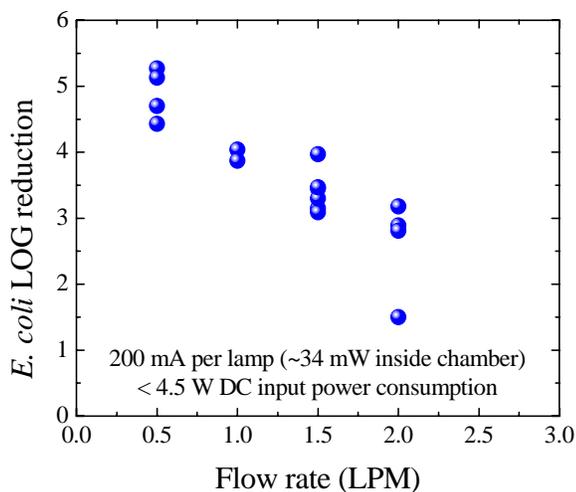


Figure 6. Efficacy of the DUV LED disinfection vs. flow rate for *E. coli* eradication.

Disinfection of viruses usually requires higher UV dosage than that for bacteria. MS2 bacteriophage from Leviviridae family was selected as representative microorganisms of viruses.

Results of the second experiment of MS2 bacteriophage disinfection testing of the DUV LED unit are shown in Table 1. In some experiments survival concentrations were determined approximately because lysis could not be clearly seen. The values of MS2 LOG reduction are represented by their lowest (least) limits.

Table 1. MS2 germicidal efficacy of the DUV LED lamp unit. Flow rate = 0.5 l/min; UV power = 34mW.

Initial conc. of MS2, PFU/ml	Survival conc. of MS2, PFU/ml	Log reduction
2.30E+01	4.00E-01	1.8
1.27E+05	3.50E+03	1.6
1.50E+02	<1	>2.17
5.30E+01	<0.1	>2.72
1.66E+0.2	<0.1	>3.22

As evident from Table 1, more than 1.7 LOG reduction of MS2 was achieved at very moderate dosage from DUV LED lamps. These preliminary experiments clearly demonstrate the efficiency of UV irradiation for virus disinfection.