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Characterization of a novel VHF-CCP for sterilization and decontamination of medical instruments

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Abstract: A novel VHF-CCP was developed for sterilization and decontamination of medical instruments. The sterilization process is a two-step process: at first, an evaporated liquid is set into the chamber, e.g. water. Afterwards, plasma is ignited either solely with the remaining water vapor or with hydrogen gas in addition. As a “proof-of-concept”, microbiological investigations are presented. To mimic worst case conditions, microbiological investigations were carried out in a so called “process challenging device”, a metal box with small slits (0.3 mm). Besides, the condensation process was investigated with a high-speed camera in combination with a distance microscope objective.

Keywords: plasma sterilization, VHF, liquids, evaporation, high-speed camera, optical emission spectroscopy, process challenging device

1. Introduction

Cleaning, sterilization and decontamination of surfaces of various instruments are key processes in the medical field and in pharmacological industry. The main objective of these processes is to remove, inactivate or destroy all pathogens in order to prevent infections, inflammations or transmission of diseases. However, the traditionally employed techniques suffer from certain limitations and are in many cases insufficient to guarantee the complete elimination of pathogens at all, or only after serious damage of the treated objects themselves. Therefore, new sterilization techniques are urgently required. One promising alternative that gained increased attention is plasma sterilization. The capabilities of plasma sterilization to remove and inactivate proteins, bacteria, bacterial spores and prions could be shown in several laboratory setups, e.g. [1-3]. Although plasma sterilization offers several advantages compared to the traditionally employed techniques, it is a challenge to sterilize small, complex metal geometries, due to shielding characteristics. To overcome this problem, a two –

step process is approached. At first, water vapor is set into the chamber. Due to the pressure difference atmospheric pressure to vacuum (10 Pa), the volume is saturated immediately and the vapor condensates on the surface. During this process, energy is released directly on the surface. After condensation, plasma is ignited, either with water vapor or with hydrogen gas which is given to the chamber additionally.

2. Experimental Setup

The presented plasma reactor is a low-pressure capacitively coupled plasma source (see **figure 1**).

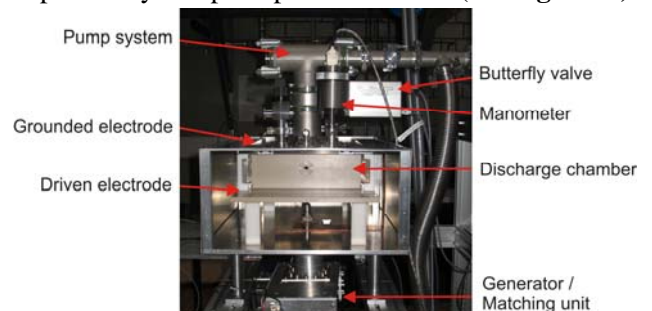


Figure 1. *Experimental set-up*

The plasma chamber has an inner size of 320 mm x 220 mm. The gap between the grounded and the driven electrode is 80 mm. Both electrodes have the same size as the chamber.

The discharge chamber itself is composed of PEEK, a high-performance plastic, and shaped like a drawer as depicted in **figure 2**.

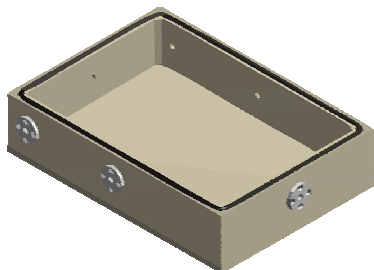


Figure 2. Discharge chamber of the VHF-CCP

The drawer is easy and safe to handle, thus designed to meet industrial needs. It is connected to a rotary vane pump and a gas inlet system through a gas shower with 16 bores inside the grounded electrode to achieve a homogeneous discharge. The gas inlet system is composed of four MKS mass flow controllers connected to Ar, O₂, N₂ and H₂. For all data presented within this contribution, H₂, O₂, or a combination of both was chosen as process gas with a constant total gas flow of 20 sccm and a pressure of 5 Pa, or 10 Pa respectively.

To enhance the plasma sterilization process a two-step process is approached, inasmuch as evaporated water is given into the chamber. Therefore, an evaporator is attached to the chamber, fed by a syringe drive (PSD/4, Hamilton®). The syringe drive allows providing small amounts of water in a reproducible manner. The amount is tunable in the range of 1 – 25 ml. Furthermore, the drive speed of the syringe is tunable, thus the pressure of the liquid reaching the evaporator.

The rf plasma source is connected to the lower electrode. It operates at a frequency of 81.36 MHz with a possible maximum power output of 500 W. It is matched to the discharge through a variable matching network in T-type configuration.

2.1 Diagnostic methods

The plasma was investigated by optical emission spectrometry. The optical emission spectra were recorded through a quartz window using a relative and absolute calibrated [4] Ocean Optics QE65000 in the spectral range of 200-950 nm with a spectral resolution of 1.3 nm.

The condensation process was investigated with the high speed camera (Photron® Fastcam MC 2.1). The camera has a 24 bit CMOS sensor with 10µm pixel.. The highest frame rate is 10 000 fps, with a resolution of 512x96 pixel, or 2000 fps at full image resolution (512x512 pixel). Attached to the camera we used a high magnification zoom lense (Navitar® 12X Zoom). The measurements presented within this contribution were carried out with 500 fps at highest resolution of 512x512 pixels.

2.2 Biological samples

The experiments presented here focus on spores of *B. atrophaeus*, defined by the American Type Culture Collection (ATCC) as ATCC 51189. The spores were provided and analyzed by the Fraunhofer Institute for Process Engineering and Packaging IVV in Freising.

The spores are sprayed on glass slides with an average of $1.9 \cdot 10^7$ CFUs over a loaded area of 4 cm². For each data point including the reference, three samples are treated. Treated substrates are washed with a defined amount of Ringer solution and sent back to IVV for the analysis, where a dilution series is made. The dilution is pipetted on agar plates where the colonies can be counted after a few days of incubation. Each colony represents a cell able to germinate. The result can be given in CFUs left on the holder or as the reduction of CFUs. CFUs are compared with a reference which undergoes the whole procedure except for the treatment. This value is given on a logarithmic scale as the logarithmic reduction log R:

$$\log R = \log\left(\frac{BC}{CFU}\right)$$

BC: Basis contamination
CFU: colony forming unit after treatment.

3. Results

3.1 Characterization with OES

One of the most striking effects of low-pressure plasma sterilization is the (V)UV-radiation produced by the plasma. It is well known that the wavelength between 200 and 300 nm plays a major role during the sterilization process (e.g.[1]), especially during short treatment times. Therefore, a spectrum in a pure hydrogen discharge at 5 Pa was recorded as depicted in **figure 3**. The H₂ (a-b) continuum was fitted to the measured spectrum.

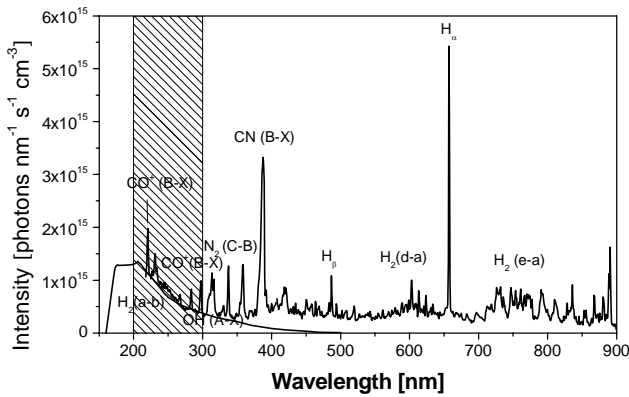


Figure 3. H₂ spectrum with fitted H₂ (a-b) continuum

From the graph we can see that the hydrogen discharge provides a high amount of radiation in the UV and particularly in the UVC range. Since the spectrometer used is absolutely calibrated, we can determine the intensity of photons per second and cm³ in that region, which is shaded in **figure 3**, to:

$$I_{H_2(a-b)200-300nm} = 7.54 \cdot 10^{16} \frac{\text{photons}}{\text{cm}^3 \text{s}}$$

Correspondingly, the spectrum of a pure water discharge was measured as shown in **figure 4**. From the graph we can see that the intensity with 4 ml water as discharge agent is around 5 orders of magnitude lower than with hydrogen as process gas. This is explainable by the fact that hydrogen is provided with a constant flow of 20 sccm whereas water is provided once with 4 ml. Another indication in favor of this approach is that the radiation depreciates with time, as can be seen in the comparison of the spectrum after 48 s plasma and

with the spectrum acquired after 120 s plasma treatment. Besides, we can see from the spectrum the Balmer series and OH (A-X), and little traces of CO.

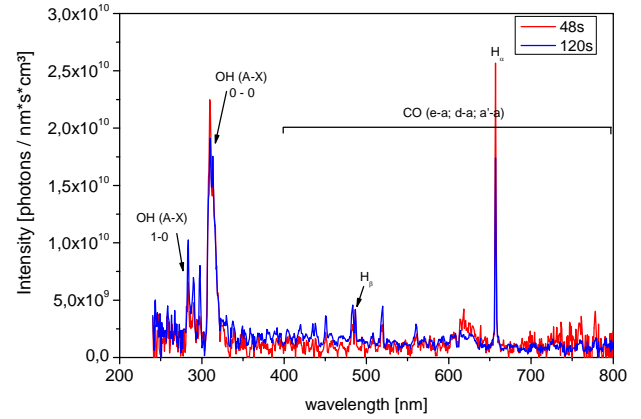


Figure 4. H₂O spectrum after 48s (red) and after 120 s (blue)

However, the spectrum of the water discharge shows that all important sterilization agents can be produced by water: UV radiation, high intensity in the UVC range, OH. On the other hand is the total amount of radiation less. The next step would be to combine both and to use the condensation energy as further sterilizing agent.

3.2 Investigation of the condensation process

In order to optimize the condensation process and to achieve a homogeneous coverage of surfaces to be sterilized, we investigated the condensation process with a high speed camera (see **figure 5**)

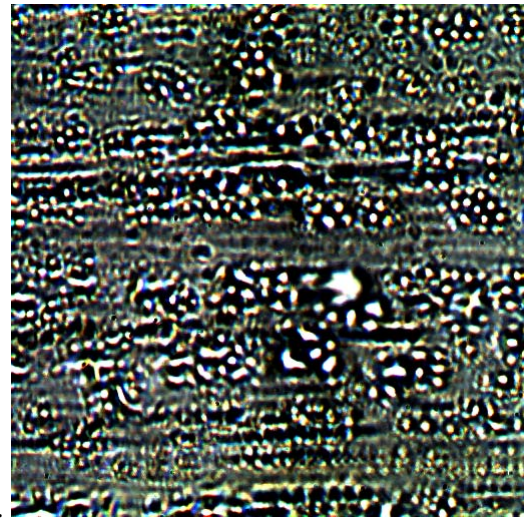


Figure 5. Image of droplets after condensation with 4 ml water

Several process parameters have been investigated, e.g. amount of evaporated water, the drive speed of

the syringe drive and the base pressure. As an example the influence of the base pressure is discussed in this contribution. **Figure 6** presents data relating to the effect of different base pressures at fixed evaporation temperature (120°C), fixed drive speed (4s for full stroke) and an amount of 2 ml.

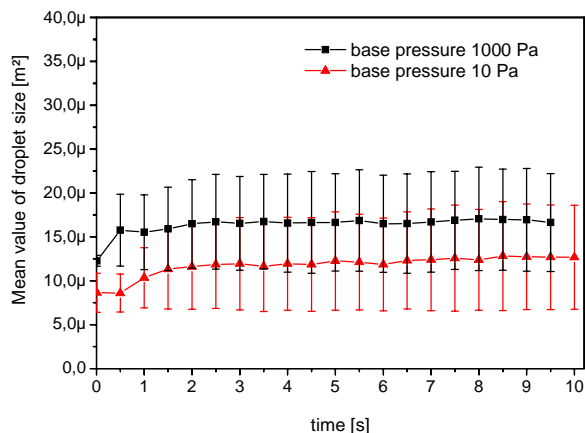


Figure 6. Droplet size after condensation with base pressure 10 Pa (red triangles) and 1000 Pa (black squares)

During the first second, the droplet size rises. This can be explained by the fact that there is a homogeneous condensation at the beginning, since the volume is oversaturated. Afterwards the heterogeneous condensation starts, with a condensation on already existing surfaces. After 1 s, the droplet size grows only marginally. At the beginning of condensation, the droplets at a base pressure of 10 Pa have a size of 8 µm, drawing level with 12 µm. In comparison, the droplets at a base pressure of 1000 Pa have a size of 12 µm during condensation start and reach a size of 17 µm after 10 s. It can be observed that the error bars soar with increasing time. That means that we achieve a non-homogeneous distribution of the droplet size, due to coalescence of the droplets. On account of that, uncovered areas grow. Taking into account the size of bacterial spores with around 1 µm, we can state that a smaller droplet size leads to a better coverage of the bacteria.

3.2 Microbiological tests

As a proof-of-concept, microbiological tests were carried out with *B. atrophaeus*. At first, 4 ml of water was given into the chamber and allowed to condensate. Afterwards, a hydrogen plasma was

ignited at 10 Pa and 400 W for 8, 16, 32, 64, 128 and 256 s. Half of the samples were placed inside a process challenging device (**figure 7**), the other half were exposed directly to the plasma.



Figure 7. Process challenging device: 3 metal boxes with each 3 slits (0,3 mm, 0,5 mm and 0,7 mm)

The results of the sterilization process are depicted in **figure 8**. Inside the PCD (0,3 mm slits) the sterilization needed more time than outside, but sterilization is possible within 256 s.

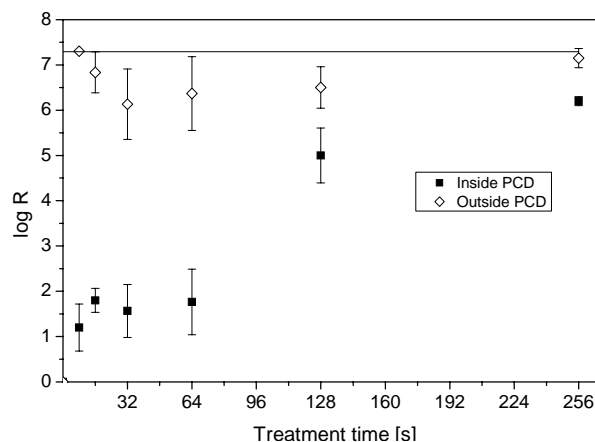


Figure 8. Sterilization tests with *B. atrophaeus* inside and outside the PCD

Conclusion

A novel VHF-CCP for sterilization and decontamination of medical instruments was presented and characterized. Optical emission spectroscopy revealed a high intensity in the for sterilization purposes important range of 200-300 nm. A two-step process was introduced to enhance sterilization for small, complex metal geometries. As a proof of concept biological tests were carried out with *B. atrophaeus*. After 256 s sterilization was achieved even inside the PCD.

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