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Edited by G. D. Megersa, E. Maťaš, J. Országh, P. Papp, Š. Matejčík

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EMISSION SPECTRA OF TRANSIENT SPARK WITH ELECTROSPRAY

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Transient spark (TS) discharge, generate low-temperature plasma rich in ions and electrons, initiating a chain of reactions that produce gaseous molecules like NO, NO_2, and HNO_2. When these gases dissolve in water, they form reactive species, including NO_2^-, NO_3^-, H_2O_2, and $ONOO^-$/ONOOH, which have potential applications in environmental processes. The solvation of these gases is enhanced by converting bulk water into electrospray (ES) microdroplets directly within the TS discharge zone. Moreover, the simultaneous production of TS and ES increases the concentration of iron ions due to electrode sputtering. This study investigates the precursor ions and atoms involved in reactive species generation using emission spectroscopy.

1. Introduction

The transient spark (TS) is a DC driven self-pulsing discharge characterized by a short-duration, high current pulses [1–4]. TS generates high-energy electrons, which collide with air molecules and atoms, causing ionization and excitation. The resulting ionized and excited species interact to form new molecules collectively referred to as reactive oxygen and nitrogen species (RONS). These gaseous RONS can dissolve upon contact with water, rendering the water "activated."

TS is able to operate in contact with water in a form of microdroplets. These microdroplets are generated by pumping the water through the electrode, on which simultaneously the transient spark is generated. Strong electric fields leads to the formation of water micro-droplets. This method of contacting the water with plasma ensures an efficient transfer of RONS into the water. [8, 9]

The primary objective of the presented study is to investigate the short lived precursors of stable RONS (such as NO, NO_2, and HNO_2) and to better understand the processes involved in their formation. To analyse the evolution and lifetimes of the relevant ions, atoms, and excited molecules, we employed both time integrated and time-resolved optical emission spectroscopy.

Although TS without ES was already studied [1-4], recent findings suggests, that the TS operated with ES might enhance the electrode erosion, introducing Fe+/Fe species into the treated water [5]. For this reason we focused here Fe+/Fe emission lines. Besides, we also studied the Hα line at 656nm, influenced by the Stark-broadening. This line can be used to determine the electron density of plasma. [3], [6] For this purpose, the FWHA of the Hα line was used according to Gigosos et al [6]. The electron density of the plasma is a key parameter for the assessment of the plasma reactivity. [3]

2. Experimental methods

Figure 1 presents a simplified schematic of the experimental setup. A stainless steel dispensing needle, serving as the anode, was connected to a high-voltage (HV) power supply delivering 12–15 kV. The grounded electrode consisted of an M5 stainless steel screw, with the electrode gap set at 9 mm. Electrospray (ES) was produced by continuously injecting deionized water through the HV needle at a flow rate of 500 µL/min using a syringe pump. A synthetic air flow rate of 1 L/min was maintained throughout the experiment. The anode voltage and discharge current were monitored using an HV probe and a current probe, respectively, with electrical signals recorded by a digital oscilloscope (Tektronix MDO34 500 MHz). Time-integrated optical emission spectra were collected using a compact

two-channel spectrometer (Ocean Optics SD2000), while time-resolved spectra with high spectral resolution were captured using a 2 m spectrometer (Carl Zeiss) coupled to an iCCD camera (Andor iStar).

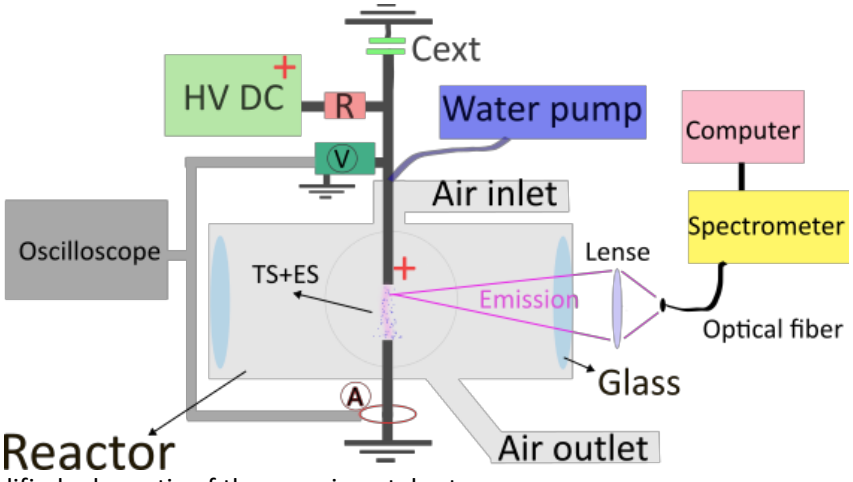

Fig. 1. A simplified schematic of the experimental setup

3. Results and Discussion

Figures 2 and 3 show the normalized emission spectra of TS with and without ES, recorded with the two-channel spectrometer. The prevalence of N^+ and O^+ lines in the spectra indicates a high degree of ionization during the high-current pulse of the TS. Alongside the N/N^+ and O/O^+ lines, the spectra also feature the Hα line and emission bands from the second positive system (SPS) of molecular nitrogen. The differences in the relative intensities of the oxygen and nitrogen lines in these figures between the TS with and without ES can be explained by the effect of frequency change. The generation of TS current pulses with ES is influenced by random droplet accumulation on the electrodes, causing more chaotic discharge behaviour, with lower frequency at the same mean discharge current (1.5mA). As it was shown by *Janda et al.* [4], the frequency of TS can have an impact on the emission spectra. However to correlate the influence of the ES on these emission spectra, further research is needed.

Figure 2. demonstrates a notable increase in the intensities of Fe^+/Fe emission lines in the TS with ES compared to TS without ES, supporting the hypothesis that electrospray enhances electrode erosion. [5] The presence of iron in the water can initiate Fenton-like reactions, leading to the gradual depletion of dissolved H_2O_2. While this reduction in H_2O_2 concentration may impact PAW composition, it could offer advantages for biomedical applications. [7] However, to fully understand the reactions, or the presence of these iron ions (or its oxides), further investigations of the PAW content is necessary, while also investigating the relevance and/or potential benefits on antibacterial properties.

We also examined the evolution of Hα line in time. Figure 4 shows example time-resolved spectra synchronized with the rising slope of the TS current pulses, measured by the Carl Zeiss spectrometer with the iCCD camera. From multiple samples, using python fitting program to fit the Voigth profile function, we obtained the FWHA and electron density from the following equation [6]:

$$\Delta\lambda_{FWHA}^{H\alpha} = 0.549\ nm\ (\frac{n_e}{10^{23} m^{-3}})^{0.67965}, \quad (1)$$

where $\Delta\lambda_{FWHA}^{H\alpha}$ is the FWHA (in nm) of the Hα line and n_e is the electron density.

As can be seen on Figure 5., the calculated electron density decreases over time. The first measured spectrum is approximately 20ns delayed compared to the start of the current rise, due to delay of the triggering signal sending to the iCCD camera. That means, the electron density of the first few ns can be higher. Nonetheless, these obtained results of high electron density (≈10^{18} cm^{-3}) prove a high degree

of ionization and high reactivity of the plasma generated by the TS discharge, showing that there is no significant difference between with and without ES, from this point of view.

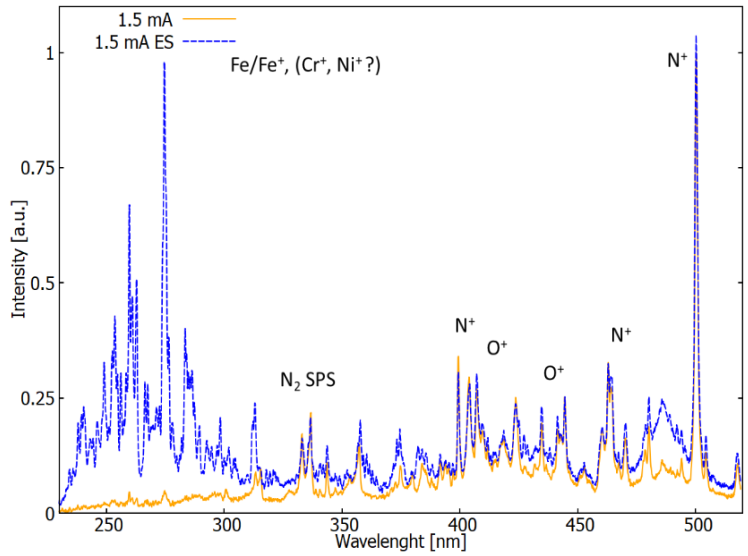

Fig. 2. Normalized time-integrated UV-VIS emission spectra of TS w/wo ES at mean current of 1.5 mA.

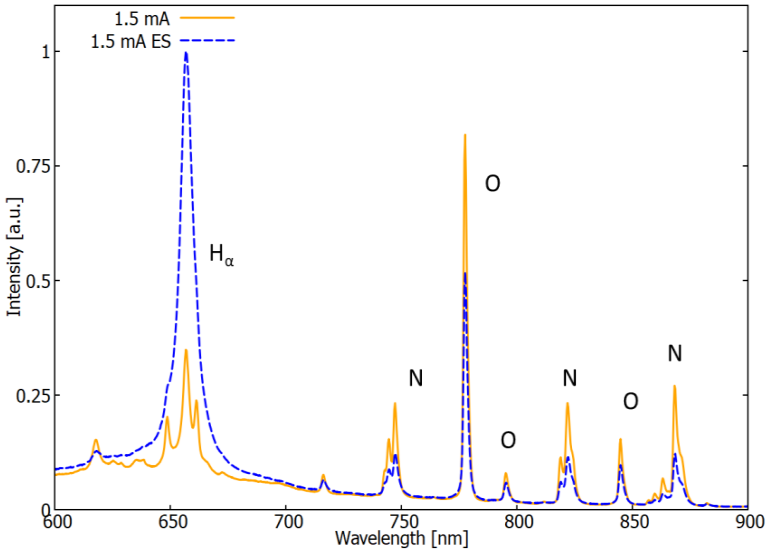

Fig. 3. Normalized time-integrated UV-VIS emission spectra of TS w/wo ES at mean current of 1 mA.

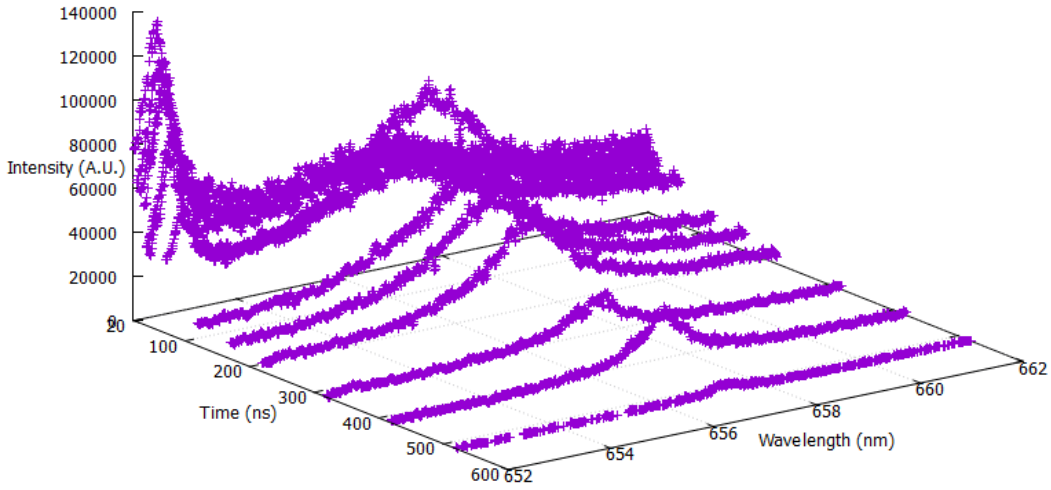

Fig. 4. Time-resolved emission spectra of TS with ES, showing Hα line evolution

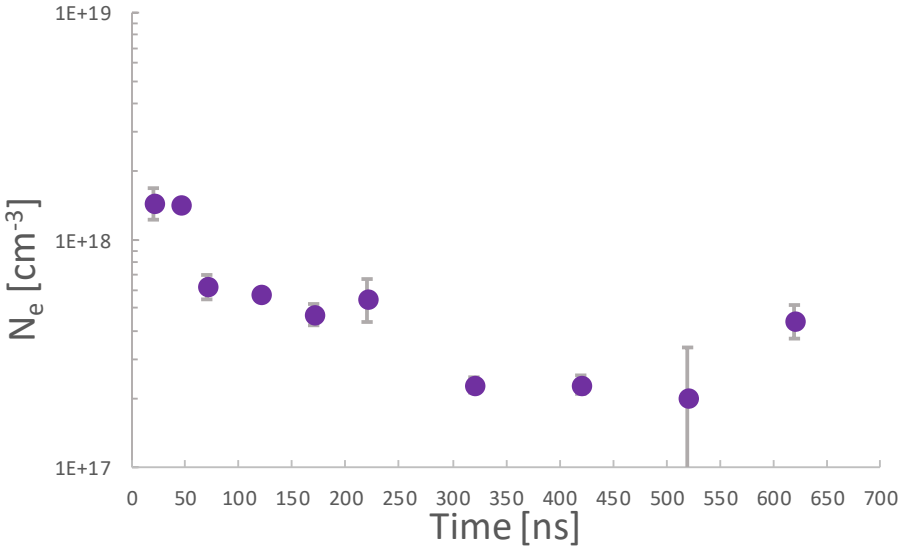

Fig. 5. Evolution of electron density in time for TS with ES, derived from the Stark broadening of the Hα emission line.

4. Conclusions

The emission spectra of TS discharge w/wo ES were dominated by N^+ and O^+ lines, as well as N, O atoms, and N_2 SPS emissions. These findings confirm the high degree of ionization and dissociation in the plasma, however the specific influence of ES on the emission spectra warrants further detailed investigation. Time-resolved spectra of the Hα line, were used to calculate the electron density, again confirming the plasma's high ionization level and reactivity. In the presence of ES, a marked increase in iron ion emission line intensities was detected, attributed to enhanced electrode sputtering.

In further research, we plan to investigate the time resolved spectra of the nitrogen, oxygen and iron ions observed in the integrated emission spectra. These studies aim to elucidate the processes and reaction pathways of plasma chemistry. Additionally, we plan to examine the influence of various parameters and conditions, such as electrical parameters or air- and water flowrate, on the emission spectra. Further research is also needed to identify the types of iron species from electrode erosion, as well as to evaluate the relevance in bio-chemistry of the PAW, and possibly examine other metallic materials as electrode.

Acknowledgements:

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