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Optical Diagnostics of Micro Discharge Jets

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Micro plasmas operated at ambient pressure with dimensions of the confining geometry in the order of a few ten micrometers to a millimeter are actually in the focus of interest due to the broad regime of applicability they offer and due to a similarly broad range of open physical questions. Here we present optical measurements within the discharge core and the effluent region of an especially developed micro discharge jet. To get an understanding of the complex system of this discharge it is important to analyse transport phenomena of energy and particles within both parts of the discharge by various highly sophisticated diagnostics. As a consequence of the limited access and the dimensions of the micro discharge most of these diagnostics are optical. Here we present diagnostics applied to determine spatially resolved absolute atomic oxygen densities as the most reactive constituent of the effluent, density maps of ozone as final reaction product of the gas chemical chain induced by the discharge and phase resolved optical emission spectroscopy yielding insight into the excitation dynamics of the discharge.

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1 Introduction

Non-thermal non-equilibrium atmospheric pressure plasmas recently have drawn considerable attention due to their enormous potential for technological applications [1, 2].

A common feature is the reduced spatial dimension of the confining structures, e.g. electrodes, stabilising the discharge and preventing the transition to a "thermal" arc discharge. Typical dimensions vary from a few microns up to a few millimetres. The wide range of these discharges is, therefore, often sub-summarised under the topic "microplasmas" [3–6].

High concentrations of radicals and a low gas temperature, suitable for many applications especially in modifications of sensitive surfaces, can be provided by jet-like devices without the requirement of complicated and expensive vacuum systems. Many of these discharges operate in a mixture of a noble gas, preferably helium, and a small molecular component - depending on the application envisaged. Despite enormous potential for technological applications, fundamentals of non-equilibrium plasmas at ambient pressure are only rudimentarily understood.

The discharge concept adopted here is the Atmospheric Pressure Plasma Jet (APPJ) introduced by Selwyn, Hicks and co-workers [7] in 1998. This technically relatively simple capacitively coupled device is typically operated at an excitation frequency of 13.56 MHz and an electrode spacing of about 1 mm. The length and width of the devices are usually in the order of a few centimetres. A mixture of noble and molecular gas is flushed through this discharge area at a high flow rate in the order of m^3h^{-1} . Typical molecular admixtures are in the order of 1 %. The atomic radical concentration in the effluent leaving the discharge 'core' is typically in the order of 10^{15} cm^{-3} [8]. These radicals are assumed to be responsible for the surface modifying properties of the jet.

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Many experiments demonstrating the surface modification abilities of the APPJ have been performed by Selwyn and co-workers [9–11].

One of the phenomena still discussed for these discharges concerns the range of stability. At low powers the discharge is characterised as a typical α -mode discharge, while at higher powers it suddenly turns into γ -mode [12]. The discharge then becomes inhomogeneous showing bright concentrated discharge columns which can even become ohmic. The distinction between these two modes is not precise in literature. In general this condition is designated as "arcing mode". The stability range over which the discharge is operating in the homogeneous α -mode is reduced significantly e.g. if the base gas is changed to argon [13]. The mode transition and the corresponding stabilisation of these discharges along with the related structure formation processes are extremely complex and a major issue in understanding the fundamental discharge mechanisms. For the build-up of an instability it is essential to examine the possible energy transport mechanisms within the discharge. These are influenced by many properties of the discharge, such as the plasma boundary sheath dynamics, plasma wall interactions influencing the production of atoms, transport of particles and radiation through the discharge, etc. Figure 1 gives a sketch of the complex system combining discharge core, the effluent, and finally the surface to be treated that has to be understood for e.g. improvement of the efficiency. Only a few approaches on the mode transitions from the modelling and simulation side, based on drift-diffusion approximation in a fluid picture, have been presented recently [14, 15].

Another open question is the range of application of the discharge. It was observed that even 10 cm outside the discharge core emission of excited atomic oxygen can be detected perpendicular to the direction of the effluent [13]. These distances are much larger than can be expected according to the natural lifetime. The energy transfer processes providing this excited atomic oxygen are still being discussed. Possible explanations such as excitation by helium or argon metastables or radiation are still not convincing.

Fig. 1 Scheme indicating the complexity of the diagnostic problem of the combined system of discharge core and effluent.

The experiments carried out clearly revealed that in order to understand the complex energy transport mechanisms in an APPJ, the connection between the effluent and the discharge core is of major importance. The investigation of the discharge dynamics and transport processes within the discharge volume is, therefore, essential and the measurement of absolute densities of atomic oxygen and ozone in the discharge region and the effluent provides important information.

In the following we present a selection of measurements providing information on a limited sub-set of the above mentioned constituents of a plane parallel APPJ and an especially designed μ -APPJ. Diagnostics employed range from optical emission spectroscopy (OES) in the UV to two-photon absorption laser induced fluorescence spectroscopy (TALIF) applied for the measurement of atomic radicals in the effluent. Knowledge of the concentration profile of ozone is indispensable for the understanding of energy transport processes in the effluent. This is accessed by UV absorption spectroscopy. Phase resolved optical emission spectroscopy (PROES) yields insight into the discharge dynamics, in particular electron heating mechanisms and plasma boundary sheath dynamics. This allows a direct comparison to simulation results.

1.1 Micro atmospheric pressure plasma jet (μ -APPJ)

Measurements in the effluent of an APPJ are obviously without severe geometric limitations. In contrast, diagnostic access to the discharge core of the standard coaxial APPJ - as is the case for many microdischarges - is not possible due to electrode configurations. Even for the plane parallel version applied here the access to the discharge volume is restricted due to the severely limited observation angle determined by the aspect ratio of 1 mm electrode distance compared to 40 mm electrode width. For these reasons investigations on discharge

phenomena have been mainly concentrated on electrical characterisation of the discharge [16, 17]. Application of probes is prohibited correspondingly.

Hence a specifically modified micro APPJ (Fig. 2) with reduced discharge volume of about $1 \times 1 \times 30 \text{ mm}^3$ (μ -APPJ) was developed that provides excellent diagnostic access to the discharge volume and the interface to the effluent region, in particular for optical techniques. This allows detailed investigations of the discharge dynamics and energy transport mechanisms from the discharge to the effluent. Figure 2 shows a sketch of the μ -APPJ and the diagnostic access. The operation parameters of the μ -APPJ are scaled down from the standard jet - where possible - to keep the devices comparable. Typical transceiver powers for this device range in the order of 5 to 20 W.

Fig. 2 Photograph of the nozzle of the μ -APPJ (a) and a sketch of the possible diagnostic access (b). (Online colour: www.cpp-journal.org).

In conclusion, the developed μ -APPJ is ideally suited for detailed investigation of the discharge dynamics by combining a multitude of diagnostics and adapted modelling and simulation approaches.

2 Time-integrated optical emission spectroscopy in the UV and VUV

Spatially resolved time-integrated optical emission spectroscopy in the visible and the (vacuum) ultraviolet yields valuable information on gas composition, temperatures and radiation transport processes in both, discharge and effluent. It is especially well suited for micro discharges since only one window for optical access to the emitting plasma is required. Quantities accessible from the detected radiation are e.g. the gas temperature from rotational bands of molecular components or for high enough levels of ionisation the electron density from the Stark broadening of atomic lines [18]. Here, we present measurements of the ultraviolet (UV) and vacuum ultraviolet (VUV) emission of the APPJ in search for an energy transport mechanism providing the energy for excited atomic oxygen far outside the discharge. Helium and argon metastables can be excluded since they are subject to very fast two-step destruction processes. During this process radiation in the deep vacuum ultraviolet is emitted (e.g. argon excimer continua). This radiation in the UV and VUV wavelength range has to be taken into account as an energy transport mechanism for the excitation or production of excited atomic oxygen outside the discharge.

The experiment sketched in figure 3 a) was set up to evaluate the extension of this radiation under realistic conditions. The entrance slit of an evacuated 25 cm monochromator (Minuteman 302 VM) is positioned directly in front of the nozzle of the plane parallel APPJ. The discharge with water-cooled aluminum electrodes of $9 \times 4 \text{ cm}^2$ and an electrode gap of 1.2 mm is installed on a stepper motor controlled translational table aligned with the entrance slit. The slit is closed vacuum tight by a 1 mm thick MgF_2 window. For the detection a solar blind photomultiplier (EMI G26-315) with CsTe photocathode is installed to the exit port.

During the continuous operation at 100 W transceiver power, $1 \text{ m}^3/\text{h}$ flow of helium with a minority gas of 0.5% of oxygen the discharge is moved away in steps of 5 mm from the starting position at 5 mm distance to the entrance slit. At each position a spectrum is recorded between about 115 and 320 nm. Figure 3 b) shows the result of such a scan for a helium based measurement. As can be seen easily the radiation is transported over several centimeters into the surrounding atmosphere. At this point it has to be realised that the radiation is probably transported in the jet's own atmosphere. The main spectral features visible are the atomic oxygen line at $\lambda = 130 \text{ nm}$, the Schumann-Runge Bands and NO bands from impurities between 180 and 300 nm. Especially

the radiation at 130 nm in combination with metastable molecular oxygen might be a source of excited atomic oxygen outside the discharge core. Furthermore, the energy per photon (≥ 10 eV) hitting a sensitive surface is high enough to drive molecular processes. Regarding applications on skin an estimate of the potential risk of this radiation might be drawn from the absolutely calibrated VUV measurements carried out for a similar micro jet by Foest et.al. [19].

Fig. 3 Experimental set-up (a) for measurement of the transmission of UV radiation from the core discharge into the ambient air and (b) spectral map of the radiation determined up to 10 cm outside the jet for a helium based discharge. (Online colour: www.cpp-journal.org).

3 Two-photon absorption laser-induced fluorescence

In this section we discuss quantitative, spatially resolved two-photon absorption laser-induced fluorescence (TALIF) measurements of atomic oxygen ground state densities in the effluent of the μ -APPJ operated with O_2/He . Titration with a flow-tube reactor is commonly applied as a reference source for calibration [20]. Recently it was shown that a titration scheme allows a spatially not resolved measurement of the atomic nitrogen concentration in the effluent of a similar discharge [21]. The results presented here are based on a TALIF calibration scheme with xenon as described in greater detail in [8]. The method is based on comparative TALIF measurements using xenon as noble gas as a reference with a two-photon resonance spectrally close to that of the atomic oxygen to be quantified [22]. Since TALIF has already been described elsewhere [23] we concentrate here on the most critical points particularly considering atmospheric pressure. Several rules have to be observed: the conditions of excitation and detection should be as similar as possible for both two-photon transitions; this includes the properties of the laser output and the optical set-up. Only the unsaturated quadratic signal response allows a quantitative comparison. Various saturation effects as ground state depletion exhibit rather complex intensity dependencies with individual thresholds. Artificial particle generation, e.g. by photo-dissociation has to be mentioned here explicitly due to its particular importance for mixtures with molecular components. In conclusion the laser intensity has to be kept as low as possible. The unknown atomic ground state density n_O is then related to the reference gas density n_{Xe} by:

$$\frac{S_O}{S_{Xe}} = \frac{n_O}{n_{Xe}} \frac{\sigma_O^{(2)}}{\sigma_{Xe}^{(2)}} \frac{T_O \eta_O}{T_{Xe} \eta_{Xe}} \frac{a_O}{a_{Xe}} \quad (1)$$

with the fluorescence signal S (integrated over fluorescence wavelength, excitation wavelength and interaction time), the effective branching ratio a of the observed fluorescence transition, the transmission T of the detection optics and the detector's quantum efficiency η for the fluorescence wavelength, and the two-photon excitation cross section $\sigma^{(2)}$.

At atmospheric pressure collisional quenching (collisional de-excitation) becomes important if not dominant reducing the effective branching ratio of a spontaneous transition $i \rightarrow k$:

$$a = \frac{A_{ik}}{A_i} \quad (2)$$

where A_i denotes the effective decay rate including quenching:

$$A_i = \sum_k A_{ik} + \sum_q k_q n_q \quad (3)$$

Here, it becomes obvious that knowing the relevant quenching coefficients k_q and the densities of the corresponding quenching partners n_q is essential. The majority species in the jet effluent are the feed gases helium and molecular oxygen. They also represent the dominant colliders responsible for the quenching of the laser-excited oxygen atoms. We can infer the effective quenching rate on the basis of measured quenching coefficients, since we know the dominant colliders responsible for quenching of the laser excited oxygen atoms as well as their density distributions. Under the conditions investigated molecular oxygen is the dominant quenching partner [24].

The local effective quenching rate can be calculated on the basis of the measured room temperature quenching coefficients and the measured gas temperature field shown in figure 4 (a). The gas temperature was measured here by a thermocouple. The density distributions of both colliders are simply related to the gas temperature according to Dalton's law and assuming a constant ratio of molecular oxygen to helium throughout the jet's effluent.

In the following we discuss absolute measurements of atomic oxygen in the effluent region of an μ -APPJ operated in helium with a 1% admixture of molecular oxygen; the experimental setup is in general identical to the one presented in [8] and is therefore omitted here. As major change the vessel is replaced by a smaller one taking into account the drastically reduced gas flow of the μ -APPJ. The vessel is necessary for absolute calibration using xenon as filling gas.

The determination of the absolute atomic density is performed in two steps. Primarily, a two-dimensional fluorescence map of the jet effluent is recorded, where the on-resonance fluorescence signal is measured as a function of the axial and transversal positions. Then, the calibration measurement is performed at one spatial position. The analogous procedure is repeated for the case of xenon filling at the close-by Xe resonance. Finally the fluorescence map data are converted into spectrally integrated data using the measured temperature distribution. The resulting map of the absolute atomic oxygen density for a 1.4 slm helium flux, a 1% O₂ admixture and an RF power of 15 W transceiver power is shown in figure 4 (b). The effective radial resolution is of the order of 2 mm. The atomic oxygen density and the gas temperature are highest close to the nozzle: $8 \cdot 10^{13} \text{ cm}^{-3}$ and 35°C , respectively. The atomic density decreases by a factor of about five over a distance of about 4 centimeters. The maximum values for the APPJ as measured by Niemi et al. [8] close to the nozzle are about one order of magnitude higher. To compare both measurements it has to be taken into account that due to the electrode construction the zero position of the actual measurements is 5 mm further away from the discharge nozzle. Another effect might be the increased influence of the quartz walls of the μ -APPJ that are much closer to the discharge center than is the case for the APPJ. In conclusion we assume that the oxygen production of the μ -APPJ is comparable to that of the APPJ as expected by scaling down the operational parameters.

An important fact is that the atomic density in this helium atmosphere remains sharply concentrated to the beam axis showing only a slightly larger beam divergence than the temperature. The observed distribution map of ground state atomic oxygen correlates reasonably well with the observation that fluorescence of excited atomic oxygen extends several centimeters outside the jet. Radiation in the UV and VUV wavelength has then to be taken into account as a second energy transport mechanism for the excitation or production of excited atomic oxygen outside the discharge.

4 Ozone concentration from UV absorption spectroscopy

In this section we describe UV absorption experiments on ozone in the effluent. Ozone is primarily created during the collision of an oxygen atom and an oxygen molecule. The high reaction rate of this process limits the range over which the desired atomic oxygen can be used for surface treatments. Under controlled conditions it can also be used to determine the amount of atomic oxygen leaving the plasma source by absolute measurement of the increase of ozone outside the nozzle. For these calculations it is important to control the gas volume of the effluent. Park and co-workers managed to reach this goal by flushing the effluent through a tube of the identical cross-section as their discharge [25]. For a more application oriented approach we installed the setup as sketched in figure 5. The effluent of the plane parallel APPJ is emitted directly through the ambient air and the optical system into the laboratory exhaust. The APPJ can be rotated and moved in 3 dimensions with a stepper motor

controlled translational stage. The axis of the gas flow is the z -axis, while the axis normal to the jet's plane will be denominated as x -axis. Consequently the axis in the plane perpendicular to the gas flow is the y -axis. By rotating the APPJ it is then possible to perform line-integrated measurements of the ozone concentrations in x and y direction up to z -distances from the nozzle.

Fig. 4 Maps of the gas temperature (a) and the atomic oxygen density (b) in the effluent of the μ -APPJ. (Online colour: www.cpp-journal.org).

Fig. 5 Sketch of the diagnostics assembly for UV absorption spectroscopic determination of the ozone concentration in the plane parallel APPJ.

The optical system for UV absorption consists of a mercury/argon spectral lamp as light source, a system of lenses and spatial filters and a monochromator equipped with a photomultiplier. For the selected mercury line at $\lambda = 253.7$ nm near the maximum of the the ozone absorption cross section a value of the absorption cross section of $\sigma = 1.14 \cdot 10^{-17} \text{ cm}^{-2}$ was measured by Parisse and co-workers [26]. A major problem of this free-running setup is the build-up of schlieren around the effluent influencing the optical path. This problem was overcome by an optical 4-f assembly using two spatial filters to exclude contributions from outside the selected pathway. The spatial resolution of the system inside the jet's plane is about 0.2 mm. To determine a spatially resolved map of the ozone concentration outside the discharge core the line-of-sight integrated UV absorption was determined from two transversal scans with a step-width of 0.2 cm for increasing distances up to 10 cm from the nozzle in x

and y direction. The ozone density can then be calculated in the tomographic approximation as given by:

$$n(x, y, z) = \frac{\int n dx \cdot \int n dy}{\iint n dx dy} \quad (4)$$

For the measurements presented here the discharge was operated at a gas flow of 1 m³/h of helium with an admixture of up to 50 sccm of molecular oxygen at powers of 50 to 150 W. The results of these measurements are shown in figure 6. The top row shows yz - and xz - cross sections of the concentration for the jet's plane being oriented perpendicular and parallel to the optical path. The bottom row shows xy - maps through the jet in planes at distances of 3 and 6 cm from the nozzle. These graphs allow some important conclusions to be drawn. First of all turbulence is setting in at a distance of about 2 cm from the nozzle entering the ambient air. Up to that point the cross section of the ozone flow produced by the jet stays rectangular. Beyond this point this compact flow spreads and soon transforms into a beam of circular shape (right subfigure of bottom row of figure 6). These effects diffusing the ozone and determining the interaction zone of the jet effluent have to be taken into account when comparing these results to the measurements of the atomic oxygen concentration. Figure 7 shows the on-axis ozone density and the plane-integrated ozone concentration in dependence of the distance from the nozzle for different oxygen admixtures.

Fig. 6 Ozone profiles in ambient air measured from UV absorption spectroscopy. (Online colour: www.cpp-journal.org).

The on-axis profile displays a very short increase of the ozone concentration up to a distance of about 2 cm. Then the concentration falls off from the maximum value. In contrast to these misleading on-axis profiles that do not take into account the distortion of the beam profile by diffusion and turbulence the plane-integrated profiles show that the conversion of atomic oxygen into ozone results in an increase of the ozone concentration continues over a distance of at least 6 cm. This fits very well the emission spectroscopic observations as mentioned before [13] and the TALIF measurements presented above and in [8].

5 Space and phase resolved OES

Phase resolved optical emission spectroscopy complements the other diagnostics by providing access to the dynamics of the excitation processes within the discharge core. It is based on measuring the population dynamics of excited states in rf discharges with ultra-high temporal resolution on a nanosecond time scale. Typically this dynamics is attributed to the light electrons. For the experiments presented here an optical system is used that already has demonstrated to be capable of allowing deep insight into the electron dynamics of low-pressure low-temperature discharges [27]. The modelling of the population dynamics is typically based on a time dependent collisional radiative model [28]. The influence of cascading processes from higher electronic states has been

Fig. 7 Axially resolved on-axis (a) and plane-integrated (b) ozone concentration profiles for increasing molecular oxygen admixture. (Online colour: www.cpp-journal.org).

found to be of particular importance for the temporal behaviour of the population of excited states. At atmospheric pressure, radiationless collisional de-excitation (quenching) determines the time scales of excited states. These time scales can vary strongly, especially for higher electronic states resulting in contributions through cascades [29].

The optical system as sketched in figure 8 (a) is based on a high-repetition rate gateable intensified charged coupled device camera (ICCD camera) with 576×384 pixels and a sensitive surface of $13.2 \times 8.8 \text{ mm}^2$. The μ -APPJ is imaged by an optical system with a magnification factor of 5 onto the micro channel plate amplifier of the camera providing a spatial resolution of $\sim 6 \mu\text{m}$ per pixel and a total image field of about 2 by 3 mm. Spectral resolution is provided by means of a tunable filter (CRI Instruments) with a tuning range from 400 to 720 nm and a typical width $\Delta\lambda_{FWHM}$ of about 20 nm that is installed directly in front of the ICCD camera. The phase resolved measurement is realized by gating the camera with a gate width of 3 ns synchronized to the excitation frequency of 13.56 MHz. This gate is delayed electronically towards a single fixed trigger point, usually the zero crossing of the rf voltage. After integration of the emission over a predefined time the phase is shifted towards the trigger by a defined time step - 3 ns for the experiments presented here - and so on. By this method one cycle of the excitation of about 74 ns is resolved into 25 time intervals yielding 25 images of the discharge from the respective phase position. Figure 8 (b) shows a sub-set of a time series of jet images taken wavelength-integrated. The red line indicates the spatial position analyzed for the phase-space profiles as shown in figure 9.

Fig. 8 Detection setup for PROES (a) and a set of phase resolved emission images taken close to the nozzle of the μ -APPJ on the right hand of each image for operation in pure helium. (Online colour: www.cpp-journal.org).

The experiments presented in figure 9 are carried out with pure helium as discharge gas at a flow of 1 slm and at transceiver powers of 14 and 32 W. The tunable filter is not installed yielding wavelength integrated results. The images analysed here were measured about 2 mm from the nozzle inside the discharge as indicated in figure 8 (b). From each image of the sequence the same column of the camera image corresponding to the interelectrode axis is selected. The combination of these profiles from all phase positions finally yields the spatio-temporal profiles displayed in figure 9.

From this spatio-temporal profile conclusions can be drawn on the electron kinetics within the discharge between the electrodes. Comparison of the graphs for 14 and 32 W of input power shows significant deviations giving insight into the electron dynamics. For the higher power (figure 9 (b)) a comparison with the model of Shi and Kong [15] shows a very good agreement of our measured emission structure to the calculated electron production rates of an α -mode discharge at atmospheric pressure as displayed in fig. 3 a) of that publication. The fluid model calculates the dynamics of the ionisation rate between the electrodes of an rf excited helium discharge at atmospheric pressure for an interelectrode distance of 2.4 mm. For the comparison it has to be taken into account that the phase in the model is started at the maximum negative potential. It is obvious that in the experiment two pairs of emission maxima are observable showing a very similar behaviour to that of the model. From each pair one maximum can be attributed to the sheath-expansion feature well-known from low-pressure capacitively coupled discharges [30]. The second maximum of slightly lower intensity appearing at the opposite electrode must then be attributed to electrons entering the opposite sheath collapsing at a slower velocity. The differences in the velocity of the sheaths are easily visible in figure 9 (b) and indicated by dotted lines. The velocities in both sheaths are identical within the two phases. This explanation fits very well to the observation that for lower powers (figure 9 (a)) only the sheath expansion yields a localized excitation.

Fig. 9 Phase and Space resolved emission profiles from the central region of the μ -APPJ for (a) 14 and (b) 32 W transceiver power. (Online colour: www.cpp-journal.org).

As has been shown wavelength integrated measurements already yield valuable information. More detailed information on the energy distribution of the exciting electrons and the effect on the constituents of the plasma will be gained by analysing emission of specific spectral lines. Especially the influence of the oxygen admixture on the excitation dynamics is important and will be discussed in a forthcoming paper.

6 Summary

In this contribution a selection of diagnostics was presented that give access to main constituents of the plasma-effluent system that comprises the μ -APPJ. This μ -APPJ provides in contrast to most other micro discharges an optimized access to the discharge core especially for laser diagnostics. From the complete set of constituents of the system the densities of oxygen atoms and ozone were measured spatially resolved. Furthermore, it was demonstrated that phase and space resolved optical emission spectroscopy in combination with modelling of the population dynamics yields detailed information on the electron dynamics as well as the plasma boundary sheath dynamics. A direct comparison with adapted computer simulations allows, therefore, insight into the complexity of the dynamics of power dissipation mechanisms in rf discharges.

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