Temporal evolution of electron density and temperature in low pressure transient Ar/N₂ plasmas estimated by optical emission spectroscopy

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Abstract

A recently published method for the analysis of phase-resolved optical emission spectra was extended in order to permit estimation of time-resolved electron density profiles. The previously presented method combined collisional-radiative modelling with a self-absorption method to estimate the evolution of \( T_e \) with sub-cycle time-resolution. However, it was not capable to give similar profiles for \( n_e \) as the model was insensitive to its variations. The extensions proposed in this work describe a way to also estimate the electron density with sub-cycle time resolution from the changing rates of the argon Paschen 1s states. The method was applied to a low-pressure DBD-jet operated with argon and several argon–nitrogen mixtures with up to 4% N₂. Good agreement among evaluation of \( n_e \) from changing rates of individual 1s states was observed during the collisional phase and the full-cycle temporal profile could be calculated from relative changes in light emission. Electron densities exhibited a drop for larger admixtures of nitrogen and ranged from \( 10^{17} \) m\(^{-3} \) to \( 10^{18} \) m\(^{-3} \). As assumed in a previous work, the electron temperature model worked without explicit consideration of additional processes even when N₂ affected the plasma. However, presumably due to collisional quenching by nitrogen, two argon Paschen 2p levels were found to be inappropriate for \( T_e \) estimation and had to be removed. Values for electron temperature from the remaining levels remained at a similar value as for pure argon.

Keywords: phase-resolved optical emission spectroscopy (PROES), transient plasma, rare gas plasma, electron density, collisional-radiative modelling (CRM), self-absorption

1. Introduction

Optical emission spectroscopy (OES) is an established tool and widely used for diagnostic of plasmas. Due to its non-invasive nature it is especially suited for situations with limited space where insertion of probes would induce a significant disturbance of the plasma. The recent advent of fast intensified-CCDs (ICCDs) provided emission spectra also with high time-resolution. When referring to OES with sub-phase temporal resolution the term ‘phase-resolved OES’ (PROES) is commonly used. While a wide variety of models is available for the analysis of time-integrated OES spectra, literature on quantitative analysis of PROES data is still sparse. Schulze et al proposed a method based on the measurement of the excitation rate [1]. Desjardins et al applied PROES to an atmospheric plasma by combining the individual Paschen 1s states to a single parameter [2]. In a recent work, Gibson et al used PROES in combination with kinetic plasma simulations to study the impact of different...
waveforms on an atmospheric-pressure helium/argon plasma [3]. In a previous work presented by the authors, a method was proposed which combined collisional-radiative modelling (CRM) and density estimation by self-absorption to analyse optical plasma emission of a low-pressure argon jet with sub-cycle time resolution [4]. Above publications show that plasma conditions and emission intensity can vary greatly over the discharge cycle, creating a demand for time-resolved techniques.

In a previous work published by the authors, a method to estimate electron temperatures from PROES data of a low-pressure, non-equilibrium argon discharge was presented [4]. To avoid elaborate consideration of temporal dynamics, densities of the four prominent argon Paschen 1s states were estimated from a self-absorption method instead of being modelled by the CRM [5]. Based on the obtained densities for the 1s states as well as the ground state densities, population of the higher energetic Paschen 2p levels was modelled. Experimental 2p state densities were obtained with sub-cycle temporal resolution from the observed emission spectra. For this purpose, observed emission line intensities were corrected for their individual escape-factors (see equation (1)). Comparison of the modelled and experimentally measured 2p state densities yielded the electron temperature. While the ability of the method to estimate the electron temperature of a pure argon plasma was successfully demonstrated, its lacking sensitivity for changes in electron density was considered a major drawback.

This work proposes a way to overcome this drawback by estimating the electron density from variation of absolute 1s densities in subsequent time steps (frames).

Because the important 1s states were not modelled but measured directly, it was previously speculated that estimation of $T_e$ would prove robust to disturbing factors such as traces of reactive gases. To verify this assumption, the effect of admixtures of nitrogen on the electron temperature estimation was tested in this work. To estimate $n_e$ as well as $T_e$, emission spectra of a low-pressure argon jet which was operated with pure argon and small admixtures of nitrogen were analysed in this work.

2. Experiment

2.1. Set-up

The experimental set-up used in this work was described in detail before [4], hence only the most important parameters shall be repeated here. The DBD-Jet was made from a small glass tube with an inner diameter of 4 mm which served as plasma vessel and extended into a cone shape at the gas inlet. Plasma was ignited by applying a sine-wave high-voltage with a frequency of 30 kHz between two electrodes made from aluminium-tape. From the discharge tube the plasma flow expanded into a larger glass tube which was evacuated by a turbomolecular pump and served as an expansion volume. To systematically study the impact of gas composition on the plasma parameters, the original set-up was modified by an additional mass flow controller which allowed to add small admixtures of nitrogen to the argon gas. For all presented experiments the argon flow was kept constant at 100 sccm while the nitrogen flow was varied between 0% and 4%. The pressure inside the discharge tube was estimated by laser absorption measurements similar as in [4] to be 155 Pa (1.13 torr). To provide optical access for the laser in axial direction, a small hole was located in the back electrode of the discharge tube.

2.2. Spectra acquisition

Emission spectra were taken by a Czerny–Turner monochromator with an attached intensified-CCD camera (Princeton Instruments PI-MAX4 1024f). All spectra were acquired radially in the discharge tube similar to the previous experiments. Data was acquired for 90 frames, equally distributed over the discharge cycle. For each frame the exposure time was 500 ns. A relative response calibration was performed for the set-up using a Ocean Optics HL2000 Tungsten Halogen lamp prior to measurement. Similar to previous observations in [4], light intensity was found to vary by orders of magnitude within the discharge cycle (see figure 4).

Addition of nitrogen to the driving gas gave rise to optical emission from the intensive first positive system (1PS) of $N_2$ (see figure 1). Due to the long lifetime of the radiating molecular states (few $\mu$s) [6], emission intensities of nitrogen exhibited smaller variations during the discharge cycle than argon emissions used for plasma diagnostics. As a result, $N_2$ emissions were found to disturb peak fitting of the argon emissions especially during the phases of reduced intensity.

To enhance data quality, nitrogen emission of the 1PS of nitrogen was observed (see e.g. 720 to 780 nm).
Maxwell–Boltzmann distribution among the rotational levels. Transition strengths were calculated from the corresponding Franck–Condon and Hoenl-London factors. The rotational temperature $T_{rot}$ was used as free parameter allowing the fit to adjust the band shape to the observed spectrum. Due to the long lifetime of $B^1\Pi_u(v')$, $T_{rot}$ was assumed to be a good indicator for the argon gas temperature $T_{gas}$ which was required for analysis of argon emission lines [6]. Intensities of each band were free fit parameters since they were not required for further analysis in this work. Energies for all $B^1\Pi_u(v')$ and $A^3\Sigma_u(v')$ states were calculated according to energy formulas provided in [7] using constants provided in [8, 9]. Levels of $A^3\Sigma_u(v')$ were of Hunds coupling type $a$ while for $B^1\Pi_u(v')$ a transition from coupling type $a$ to type $b$ for levels with $j > 30$ was assumed. Franck–Condon Factors were taken from [10] and Hoenl-London factors were calculated according to [11].

Emission intensities of the prominent argon 2p $\rightarrow$ 1s transition array were subsequently estimated for each recorded frame from the area of their corresponding peaks considering the modelled nitrogen background emission.

### 2.3. Estimation of temporal profiles for $T_e$ and $n_e$

The previously presented method for estimation of $T_e$ will only be discussed briefly in this work and for a detailed description the reader is referred to [4]. The model combined direct estimation of the 1s states by an established self-absorption technique with CRM of the relative argon Paschen 2p state densities [5]. The underlying CRM was based on a model by Zhu and Pu which was extended by individually resolving higher-energetic levels. For a detailed description of the processes incorporated by the model see [4, 12]. In this work, the cross sections for excitation transfer for 1s$\gamma$ $\leftrightarrow$ 1s$\gamma$, 1s$\gamma$ $\leftrightarrow$ 1s$^2$ and 1s$^2$ $\leftrightarrow$ 1s$^2$ were rescaled to match with notably higher experimental rate coefficients reported in [13].

While a total of 31 excited argon levels was considered for modelling, only optical emission from the argon Paschen 2p $\rightarrow$ 1s transition array, which is commonly found to be the most intensive for low-pressure argon discharges, was used for data analysis in the presented work. In each recorded frame, densities of all four 1s levels were estimated from their radiation trapping. The same line pairs and absorption length as in [4] were used for the self-absorption technique and with the densities of the 1s states known, relative densities of the 2p states became available from the emission intensities according to equation (1)

$$I_{2p \rightarrow 1s} \sim n_{2p}A_{2p \rightarrow 1s, \gamma}(n_{1s}, T_{1s}, l). \tag{1}$$

Here $n_{2p}$, $n_{1s}$, $A_{2p \rightarrow 1s, \gamma}$, $T_{1s}$ and $l$ denote number densities of the upper and lower level of the transition, the transitions Einstein coefficient for spontaneous emission (taken from [14]), the light escape factor, the temperature of the absorbing 1s levels as well as the optical absorption length.

The electron temperature was estimated by comparing observed relative densities of several 2p states with results of the CRM for a range of electron temperatures. In this work a Maxwell–Boltzmann distribution was assumed for the EEDF to demonstrate the method. For a discussion on the impact of non-maxwellian EEDFs see [4].

In each frame the particular 1s densities, estimated from self absorption, were fed to the CRM to calculate excitation rates and radiation trapping. From the best match between measurement and simulation, $T_e$ was estimated for each 2p level individually.

By measuring 1s state densities directly instead of solving the corresponding rate-balance equations (RBES) their densities were kept constant within the solving process of the CRM. Therefore, they were no longer affected by its input parameters (in particular $n_e$). Hence the dominating population processes of the 2p states scaled linear with $n_e$ and population ratios lost their sensitivity to variations of electron density.

However, population dynamics of the four 1s states in the collisional phase of the discharge were governed by electron impact processes. Therefore their measured absolute number densities depended directly on $n_e$. Although 1s number densities themselves reacted only slowly on changes of plasma parameters and hence were not suited for a time-resolved estimation of $n_e$, the rate of the density variation was utilized for a quantitative analysis. The underlying idea is to compare observed changing rates between subsequent frames at $t$ and $t - \tau$ with simulated changing rates from the CRM. Based on their measured density profiles the changing rate $R(t)$ for each 1s state can be described as

$$R_{mea}(t) = \frac{dn_{2p}}{dt} \approx \frac{n_{1s}(t) - n_{1s}(t - \tau)}{\tau}. \tag{2}$$

While generally a direct estimation of the rate from the density profile according to equation (2) would be possible, it was found to be very prone to noise in the number 1s densities. To circumvent this problem and reduce scattering, rates were estimated from the slope of a linear fit considering three measured density values at $(t - \tau)$, $t$ and $(t + \tau)$ (see figure 2). Theoretical changing rates $R_{sim,n_e}(t)$ were simulated...
based on measured number densities \( n_1(t) \) and \( T_e(t) \) for the respective frame. Note that no RBEs were solved for the 1s states.

Because the previous model considered 1s states only as sources and sinks for population processes of other states, modelling of their changing rates demanded incorporation of some additional processes. Atomic collisions causing population of 1s states as well as de-population due to Penning ionisation were taken from [12]. Quenching of 1s states by molecular nitrogen was considered by rate constants from [15] (Ar 1s5 and 1s3) and [16] (Ar 1s4 and 1s2).

To estimate electron densities, changing rates were calculated for several values of \( n_e \) and the value showing best agreement with observed changing rates yielded the electron density. For verification, the process was performed for the 1s levels individually.

3. Results and discussion

3.1. Paschen 1s state densities of argon

Densities of two metastable (1s3 and 1s5) and two resonant (1s2 and 1s4) 1s levels are depicted in figure 3. Similarly to previous observations, densities of all 1s states exhibited significant variations during the discharge cycle featuring decline during recombination and rise during the collisional part [4]. Low intensity of underlying argon emission lines in the ‘dark/recombining phases’ around \( \frac{1}{2} \pi \) and \( \frac{3}{2} \pi \) together with relatively constant emission from molecular nitrogen complicate reliable estimation of densities in this part (see figures 1 and 4). Hence estimated 1s densities exhibited notable scattering during the dark phases. Naturally, this effect was more pronounced when the amount of nitrogen in the feeding gas was increased. The problem was partly overcome by subtracting the superposed emission bands of nitrogen 1PS during spectra fitting in the dark phase. The gas temperature used for density calculations by self-absorption was assumed to be 350 K which corresponded to the rotational temperature found to agree best with the nitrogen 1PS emission bands.

Higher admixtures of nitrogen resulted in reduced densities of all 1s states. This trend can be explained by collisional quenching of argon 1s states by nitrogen. Similar observations were made before [17, 18].

The uncertainty of the method was estimated via a Monte-Carlo approach by calculating the densities from input data which was artificially scattered. The initial intensities were randomly scattered between 0% and ±5%. The standard deviation of the resulting densities was found to be highest in the dark phases. However, for most of the cycle and especially the collisional part standard deviation was around 10% for 1s5 and 1s3 as well as 15% for 1s3 and 1s2. Especially in the case of 1s2 it was found to rise quickly as intensity dropped in the dark parts. This may very likely be accounted to the generally low density of this state.

![Figure 3. Absolute densities of all four 1s states. For better illustration one collisional part of the discharge cycle was marked.](image-url)

3.2. Electron temperature \( T_e \)

Profiles for the temporal evolution of the electron temperature in all gas combinations are depicted in figure 5. Temperatures were estimated individually from densities of nine argon Paschen 2p states.

Data quality exhibited similar trends as number density estimations of the 1s states with reduced confidence close to the ‘dark phases’ of the discharge. Considering that densities of the 1s states directly entered \( T_e \) estimation, this was likely caused by scattering of their values.

It appears noteworthy that individual estimations of \( T_e \) from different 2p states differed when nitrogen was added to the discharge. In particular, levels 2p3 and 2p9 were found to yield very high temperatures during the collisional phases while the other levels remained at lower temperatures, similar to the value for pure argon (see figure 5). This effect became more pronounced when more nitrogen was added to the
discharge and underlines the impact of N2 on argon population kinetics. Impact of superposed 1PS emission of nitrogen can be excluded as a cause for the observed deviation since its spectrum was modelled and subtracted from the measurement. Furthermore, its intensity was largely outperformed by argon emissions during the collisional phase of the discharge cycle. Hence, it may be assumed that quenching by nitrogen affected 2p2 and 2p8 more strongly than the other 2p levels. Similar observations were made before for the Paschen 1s levels of argon where a notable variation of the quenching constants was observed for the individual sub levels [15, 16].

No individual interaction cross sections of argon 2p levels with nitrogen were available, therefore this effect could not be incorporated by the model. Higher quenching rates for 2p2 and 2p8 caused the simulated density ratios, \( n_{\text{sim}}(2p_2)/n_{\text{sim}}(2p_1) \) and \( n_{\text{sim}}(2p_8)/n_{\text{sim}}(2p_1) \), to be higher than their measured values. Higher estimations for \( T_e \) were subsequently caused by the fact that 2p1 is preferably populated by ground state excitation and therefore features a stronger increase in number density when \( T_e \) increases [19]. Hence, for 2p2 and 2p8 small population ratios could only be achieved by the presented CRM under the assumption of very high values for \( T_e \). This problem may be overcome in future works when individual interaction constants for \( \text{Ar}(2p) + \text{N}_2 \) become available.

To reduce the effect of missing quenching constants on the following calculations in this work, electron temperatures estimated from 2p2 and 2p8 were removed from the calculation of the average value of \( T_e \). The averaged value was subsequently used for estimation of the electron density.

As depicted in figure 5, no notable change in average electron temperature estimated from the remaining seven 2p states was observed when nitrogen admixture was increased. However, temporal profiles were found to vary with N2 admixture, featuring less plateau-like shapes at higher

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**Figure 4.** Temporal evolution of relative argon Paschen 2p number densities for two extreme conditions.

**Figure 5.** Electron temperatures estimated from the ratios of 2p1 and eight other 2p levels for various admixtures of N2.
additions (see figure 5). This variation was accompanied by a systematic change in overall density and light emission from 2p states (see figure 4). Their temporal profiles were found to drop significantly earlier for larger admixtures of nitrogen, resulting also in longer dark periods and a less plateau-like shape. Furthermore, overall light emission was significantly lower at higher admixtures.

The findings show that, apart from the reduced number of useful 2p levels, the presented method for electron temperature estimation is robust towards small additions of a reactive gas. This may be especially important when, unlike for nitrogen, interaction constants are unknown.

The influence of uncertainties in the argon 1s densities was tested by a worst-case evaluation, assuming a deviation by ±15%. Their effect on the results of the CRM was tested in the bright part of the discharge cycle in pure argon as well as argon with 2% N2 and found to affect the resulting electron temperatures by about 5%. Furthermore, the impact of a deviation of Te, by ±10% was evaluated in the same way. Here, the indirect effect of Te due to a change of the argon Paschen 1s densities was considered by reducing/increasing their densities by √10% at the same time (see [5]). The variation caused Te to vary by around 5%. Hence, the overall uncertainty of the estimated electron temperature is mostly defined by systematic deviation among the results from individual argon 2p states when the average value is estimated. From figure 5 they were found to scatter within approximately ±20% (not considering 2p2 and 2p8 for argon–nitrogen mixtures). However, this deviation is systematic and does not originate from scattering.

3.3. Electron density ne

Electron densities estimated from the slope of the argon 1s states for two extreme cases (0% N2 and 4% N2) are depicted in figure 6. Particular good agreement between the individually estimated densities of 1s3 and 1s4 was found during the collisional phases (Φ = π and Φ = 2π). This may be accounted to a more reliable estimation of the changing rate compared to 1s2 and 1s5 due to their higher densities and generally better data quality (see figure 3). Electron density was found to drop for larger admixtures of N2. This observation is in agreement with reduced 2p number densities in figure 4 and previous observations from Thomson scattering [20].

To estimate the impact of the input parameters on the outcome of the ne estimation the input values of Te, \( \frac{dn_e}{dt} \), and Tgas were varied by ±20%, ±10% and ±10% respectively. Variations of ne in the collisional phase were compared for pure argon as well as argon with 2%N2. Assuming a ±20% deviation for Te as a worst-case scenario, ne was found to change by approximately a factor of 3. Variation of the changing rates and Tgas revealed only small disturbances of the resulting electron density (approximately 10%) which may be neglected considering the impact of uncertainties in Te and the finite simulation step-width of ne in figure 6 which was 20 steps per order of magnitude. In future works, an improvement of the modelling of the individual argon Paschen 2p levels (e.g. by improved cross sections for electron impact excitation) may reduce the systematic scattering of Te among the individual 2p levels and significantly improve the quality of ne.

While estimated values of ne for different 1s states were in satisfying agreement during the collisional phases where electron impact was governing the population mechanics, they exhibited notable scattering during the rest of the cycle and no agreement was observed. In order to extend the estimation to the whole discharge cycle, relative changes in light emission intensity were utilized.

With the temporal profiles of ne, Te and n1s known, absolute densities of all 2p states could be calculated during the collisional phase. Dividing the calculated absolute number densities by their observed densities yielded individual calibration factors for each level. With these factors evaluated...
during the collisional phase, absolute 2p state number densities could be reconstructed for the rest of the discharge cycle. Subsequently, electron densities were estimated from comparison of simulated and absolute 2p state densities at all frames. The corresponding results are depicted in Figure 7.

4. Conclusion

This work presents an approach to estimate cycle-resolved electron density profiles in a transient low-pressure argon plasma by means of phase-resolved optical emission spectroscopy (PROES). It connects to a recent publication in which estimation of electron temperatures under similar conditions was demonstrated [4]. However, the previous model was not able to resolve changes in electron density, a drawback which was compensated in this work. Information on the temporal profile of $n_e$ was extracted from variations of the 1s levels of argon which were modelled by a collisional-radiative model based on the work of Zhu and Pu [12] and compared with experimental data. To experimentally estimate argon 1s number densities, a well-known self-absorption technique was used [5].

The method was demonstrated on a low-pressure DBD-Jet which was operated with various mixtures of argon and nitrogen. During the collisional phase of the discharge cycle, good agreement among $n_e$ estimated from changing rates of different 1s levels was observed while results were scattered during the recombining phase. To overcome this limitation, measured relative argon 2p number densities were calibrated by comparison with absolute simulated 2p densities during the collisional phase. Hence, considering also variations of $T_e$, full-cycle temporal profiles of $n_e$ could be estimated for all gas compositions. Electron densities were of the order of $10^{17}$ to $10^{18}$. Higher concentrations of $N_2$ were found to cause a decrease.

The previously presented method for estimation of cycle-resolved electron temperature estimation was found to work also when nitrogen was present. However, the levels 2p$_2$ and 2p$_8$ yielded very high values for $T_e$ when nitrogen was added to the discharge, while those estimated from the other 2p states remained at the level of pure argon. This was explained by stronger quenching of 2p$_2$ and 2p$_8$ due to argon–nitrogen collisions. Those levels should therefore be avoided when $T_e$ is estimated in argon/nitrogen plasmas.

As all presented calculations are based on estimation of argon 1s densities, good quality of this data is vital. To enhance data quality, background spectra of the reactive gas were modelled and subtracted in this work. Insufficient absorption due to low 1s number densities leads to increased noise which affects all following calculations. Hence, situations with low self-absorption pose a limit for the presented method. While under the presented conditions data became already very scattered at 4% admixture due to quenching by nitrogen and low absorption by the argon 1s levels, larger absorption lengths in more extensive plasmas likely permit application of the presented method at even larger admixtures. This should be subject to further studies.

The presented method may also prove beneficial for analysis of time-integrated emission spectra when interaction constants between rare gas and reactive admixture are unknown.

In conclusion, the presented approach improves our previous model by the ability to estimate temporal profiles for $n_e$ and contributes to the development of non-invasive diagnostic techniques for transient low-pressure argon plasmas. Furthermore, our previous approach to estimate temporal profiles of $T_e$ was found to give good estimations even when nitrogen was present in the discharge without explicitly incorporating the impurity to the model.

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