Electron number density measurements in nanosecond repetitively pulsed discharges in water vapor at atmospheric pressure

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Abstract
This article reports on experiments in a nonequilibrium plasma produced by nanosecond repetitively pulsed (NRP) spark discharges in water vapor at 450 K and atmospheric pressure. The objective is to determine the electron number density in the post-discharge, with spatial and temporal resolution, to gain a better understanding of the discharge development and chemical kinetics. Electron number densities were measured in water vapor from the broadenings and shifts of the Hα and Hβ lines of the hydrogen Balmer series and of the atomic oxygen triplet at 777 nm. For an average reduced electric field of about 150 Td, high electron densities up to \(3 \times 10^{18} \text{ cm}^{-3}\) are measured at the cathode, up to \(5 \times 10^{17} \text{ cm}^{-3}\) at the anode, and up to \(4 \times 10^{16} \text{ cm}^{-3}\) in the interelectrode gap. The high density near the electrodes is attributed to ionization enhancement and secondary electron emission due to the high electric field in the plasma sheath. In the middle of the inter-electrode gap, we show that the electron density mainly decays by electron attachment reactions. The dissociation fraction of water vapor is estimated to be around 2% in the middle of the gap.

Keywords: water vapor, nonequilibrium discharges, water dissociation, hydrogen production, electron number densities, Stark broadening

1. Introduction

Atmospheric pressure nonequilibrium plasmas in steam or in water-containing gaseous mixtures are of interest for a wide variety of applications such as the production of hydrogen [1], pollutant abatement in flue gas [2, 3], bio-decontamination [4, 5], surface treatment [6], or plasma-assisted combustion when the discharge is applied in a mixture containing burnt gases [7]. Despite several previous studies [8, 9] a detailed understanding of the chemical processes in these plasma discharges is still needed. In particular, knowledge of the electron density is necessary to develop an accurate chemical kinetic model.

Under certain experimental conditions, the electron density can be obtained by analyzing the Stark broadening of
of steel, with a 40-mm inner diameter. The reactor walls were grounded.

The repetition rate of the pulses was 10 kHz (10⁴ pulses per second), and their full width at half maximum (FWHM) was about 20 ns. The peak voltage difference between the electrodes was 14 kV. In a previous study of the NRP spark regime in air, Pai et al [13] showed that, in the middle of the discharge gap, the electric field, \( E \), is given to a good approximation by the applied voltage, \( V \), divided by the interelectrode gap distance, \( d \). Thus, the average reduced electric field \( E/n_e \) calculated by dividing the applied voltage by the gap distance can be expressed as:

\[
E/n_e = V - V_{CF} \approx \frac{(V - V_{CF})kT}{dP}, \tag{1}
\]

where \( n_e \) is the gas density, \( k \) the Boltzmann constant, \( P \) the pressure (atmospheric pressure in this study), \( T \) the gas temperature, and \( V_{CF} \) is the cathode fall voltage. If we assume that \( n_e \) can be obtained from the perfect gas law, the reduced electric field can be expressed as the last term of equation (1). Several investigators have estimated the cathode fall voltage in NRP discharges [14–16] and have obtained values of the order of 1–2 kV. We note that these values of the cathode fall voltage represent a small fraction of the applied voltage at the peak of the pulse (14 kV according to figure 2). Even at the end of the period of energy deposition (\( t = 18 \) ns, where \( V = 5 \) kV), the cathode fall voltage is still 2.5–5 times lower than the total voltage. For these reasons, it is reasonable as a first approximation to neglect the cathode fall voltage, and therefore the reduced electric field plotted in figure 2 was determined by assuming \( V_{CF} = 0 \) kV in equation (1).

In our previous work [9], the temporal evolution of the temperature in the middle of the inter-electrode gap was determined by optical emission spectroscopy (OES) of the \( \text{N}_2(\text{C}^1\Pi_{u} - \text{B}^1\Pi_g) \) transition. In the steady-state pulsing regime, the temperature was found to increase from 650 K before each pulse to about 1200 K at the end of the pulse, then to decay exponentially back to 650 K before the following pulse. The value of 650 K is higher than the temperature of the gas...
entering the reactor (450 K) because of heating by previous pulses. Since the energy deposition in the gap is very fast (20 ns), the discharge is isochoric and therefore \( n_e \) remains approximately constant during the first hundreds of nanoseconds following each pulse [17]. Therefore, \( n_e \) can be determined from the gas temperature and pressure at the very beginning of each pulse, i.e. 650 K and 1 bar. Thus, instantaneous measurements of the applied voltage (LeCroy PPE20 kV) were used to calculate the reduced electric field of the NRP spark discharges under investigation. Figure 2 presents the voltage waveform and the corresponding calculated reduced electric field in the middle of the gap, which reaches a maximum of 150 Td about 8 ns after the beginning of each voltage pulse. The current waveforms were also measured by a Pearson probe (model 6586). The voltage and current measurements were then used to determine the energy deposited in each pulse, following the procedure described by Rusterholtz et al [18]. At the peak of the current pulse, the reduced electric field in the middle of the gap is about 150 Td. The energy per pulse is approximately 1.7 mJ.

As previously presented by Sainct et al [19], the discharge starts with a spark regime characterized by a homogeneous excitation of the entire gap between 0 and 4 ns (where 0 ns represent the start of light emission), which is characteristic of a streamer-less discharge. It is followed by an 8 ns conduction phase, and by a long decay with light emission for about 200 ns.

The spectroscopic measurements are performed using a 50 cm focal length spectrometer (Acton SpectraPro 2500i) equipped with an ICCD camera (Princeton Instruments iMAX, 1024 × 256 pixels). A 1200 groove mm\(^{-1}\) grating, blazed at 300 nm, is used, with a slit width of 20 µm. The associated instrumental broadening width, determined from the FWHM of a measured He–Ne laser line, is 0.05 nm.

The light emitted by the discharge is collected through a quartz window with an integration time between 10 and 100 ns, using a two-lens setup (figure 3). With this optical setup, the magnification is 2. A 600 µm diameter optical fiber, placed at the focal point of the imaging lens, is connected to the entrance slit of the spectrometer. The imaged area with this optical arrangement is a circle of diameter 300 µm.

3. Results

The spectral emission of NRP spark discharges in water vapor is mainly comprised of lines of atomic oxygen, atomic hydrogen, and of the OH radical. We will focus on the emission of the atomic oxygen triplet (777.14, 777.41 and 777.54 nm) and of the Balmer H\(_{\alpha}\) and H\(_{\beta}\) lines (656 and 486 nm). We studied both the spatial distribution of the electron number density (section 3.1) as well as its temporal evolution (section 3.2).

3.1. Spatial distribution of the electron number density

Spatially resolved measurements of the emission lines were performed every 1.5 nm along the axis of the inter-electrode gap. As shown in figure 4, the measured H\(_{\alpha}\) line is broad near the cathode (FWHM = 1.8 nm) and much narrower (0.3 nm) at the center of the inter-electrode gap. Moreover, the FWHM is practically constant along the inter-electrode axis, except at the electrodes.

The electron density, \( n_e \), was determined from the broadening of these spectral emission lines. In general, the shape of an emission line is governed by Lorentzian (natural, resonant, Van der Waals and Stark) and Gaussian (Doppler and instrumental) broadening mechanisms. The FWHMs corresponding to the different sources of broadening are summarized in table 1, along with the associated shifts.

The four main parameters influencing the line shape are the temperature, the pressure, the electron density, and the mole fraction (H or O). Since the gas temperature ranges from 650 to 1200 K [9], the Doppler broadening FWHM has a maximum value of 0.018, 0.011 and 0.005 nm at 1200 K for H\(_{\alpha}\), H\(_{\beta}\), and O\(^*\) (777 nm), respectively. Thus the Doppler broadening width is below the instrumental broadening width of 0.05 nm, and therefore does not significantly contribute to the observed total broadening.

The van der Waals and resonant broadening mechanisms, which are pressure dependent, also affect the lineshape. In the discharge under investigation, the pressure in the plasma channel can increase for a short time as a result of ultrafast heating [18]. For an isochoric increase of temperature from 650 to 1200 K, the pressure is estimated from the perfect gas law to increase by a factor of about 2. The maximum pressure broadening FWHM is estimated to be 0.08, 0.06 and 0.03 nm at 2 bar for H\(_{\alpha}\), H\(_{\beta}\), and O\(^*\), respectively. In summary, for the H\(_{\alpha}\) line, the sum of all Lorentzian broadening widths (excluding Stark broadening) is 0.08 nm, and the quadratic average of the Gaussian widths (Doppler + instrumental) is less than 0.09 nm. When compared to the measured width of the H\(_{\alpha}\) line shown in figure 4 (0.3 nm in the middle of the inter-electrode gap and 1.8 nm at the cathode), it is clear that the dominant broadening mechanism is Stark broadening.
Figure 5 shows the H\textsubscript{α} lineshape measured at the cathode from 10 to 20 ns after the beginning of the pulse. Interestingly, the line can only be fitted by a sum of two Lorentzian components. In addition, the broad component is shifted by about 1.4 nm relative to the narrow component. To confirm the assumption that the observed broadening is mainly due to the Stark effect, the shifts of the H\textsubscript{α}-line measured at various positions and times are compared in figure 6 with three shift models from the literature: (1) the Stark shift model of Griem et al [20] at 0.8 bar for electronic temperatures ranging from 13 000 to 18 000 K, (2) the Stark shift model of Boddeker et al [21] at atmospheric pressure and for electron temperatures ranging from 65 000 to 100 000 K, and (3) the van der Waals shift model of Konjevic et al [11]. The experimental shifts closely match the Stark shifts, thus confirming that the H\textsubscript{α} lineshape broadening is due to the Stark effect and not to

**Table 1.** Broadening FWHMs and shifts (in nm) for H\textsubscript{α} at 656.56 nm, H\textsubscript{β} at 486.132 nm and the O\textsuperscript{*} triplet at 777 nm, as a function of $P$, $T$, $n_e$, $X_\text{H}$ and $X_\text{O}$. $P$ is the pressure in bar, $T$ the gas temperature in K, $n_e$ the electronic density in cm$^{-3}$, $X_\text{O}$ the oxygen mole fraction and $X_\text{H}$ the hydrogen mole fraction.

|       | $H_\alpha$ | $H_\beta$ | $O^*$ |
|-------|------------|------------|-------|
| $\Delta\lambda_{\text{Natural}}$ | $11 \times 10^{-5}$ | $6.2 \times 10^{-5}$ | $1.1 \times 10^{-5}$ |
| $\Delta\lambda_{\text{Doppler}}$ | $11 \times 10^{-5} T^{0.5}$ | $3.5 \times 10^{-5} T^{0.5}$ | $1.4 \times 10^{-4} T^{0.5}$ |
| $\Delta\lambda_{\text{VdW}}$ | $5.4 P/T^{0.7}$ | $4.1 P/T^{0.7}$ | $2.2 P/T^{0.7}$ |
| $\Delta\lambda_{\text{Resonance}}$ | $122 X_\text{H} P/T$ | $60.4 X_\text{H} P/T$ | $5 \times 10^{-3} X_\text{O} P/T$ |
| $\Delta\lambda_{\text{Stark}}$ | $3.9 \times 10^{-12} n_e^{0.668}$ | $2 \times 10^{-11} n_e^{0.668}$ | $6.5 \times 10^{-19} n_e$ |
| $\gamma_{\text{VdW}}$ | $1/3 \Delta\lambda_{\text{VdW}}^{0.5}$ | $2 \times 10^{-11} n_e^{0.668}$ | $6.5 \times 10^{-19} n_e$ |
| $\gamma_{\text{Stark}}$ | 0.01 $\Delta\lambda_{\text{Stark}}^{0.5}$ | 0 | not used |
| References | [10, 17, 20, 21] | [11, 12, 22] | [23] |

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pressure broadening. The shift of the O\(^*\) line is too small to be measured here, and the H\(\beta\) line does not exhibit a shift.

The electron densities estimated from the Stark broadened H\(_{\alpha}\)-line as a function of position along the inter-electrode axis are given in figure 7. Each data point results from an integration over 10 ns and is averaged over 10\(^6\) pulses and a spot of diameter 300 \(\mu\)m. Since the spectra obtained at −3 mm (cathode) and 3 mm (anode) could only be fitted by using two components, as shown in figure 5, we plot two values of the electron density derived from each broadening at the electrodes. The origin of the two components is discussed in section 4.

### 3.2. Temporal evolution of the electron number density

In this section, we present the temporal evolution of the electron number densities measured from the H\(_{\alpha}\) line at the cathode and in the middle of the inter-electrode gap. To add further support to the measured electron densities, we also present electron densities measured from the H\(\beta\) and O\(^*\) lines (figure 8). The shape of the H\(\beta\) line presents a dip in the centerline, due to electrostatic interactions, that is not captured by the Lorentzian...
function used for the fit. However, the Stark FWHM of the H\textsubscript{β} transition fitted with a Lorentzian function remains in good agreement (within 25\%) with the more advanced models [10]. The temporal evolution of the electron densities measured with the three lines (H\textsubscript{α}, H\textsubscript{β} and O\textsuperscript{*}) at the cathode and in the middle of the gap is presented in figures 9 and 10.

At the cathode (figure 9), the results obtained from the Stark broadening of the three lines are in good agreement up to 100 ns. After 100 ns, the electron density is below 10\textsuperscript{17} cm\textsuperscript{−3}, which is lower than the sensitivity limit when using the atomic oxygen triplet. The densities measured are up to two orders of magnitude lower than during the pulse, which leads to large error bars. The electron number density rises to a maximum during the 20 ns pulse, and then follows an exponential decay. It is important to point out that the data taken during the pulse (0–20 ns) may include the effects of the external electric field [24].

In the middle of the inter-electrode gap (figure 10), the emission from the atomic oxygen triplet is dominated by instrumental broadening and therefore cannot be used to determine the electron densities. However, the H\textsubscript{α} and H\textsubscript{β} lines can be used, and the measurements are in good agreement. Thus, these measurements confirm the results obtained with the H\textsubscript{α} line.

4. Discussion

4.1. Electron density close to the electrodes

Several authors have previously observed multicomponent H\textsubscript{α} or H\textsubscript{β} lines in spark discharges, but the reasons have not been clearly explained in general. Some studies have attributed them to high energy hydrogen ions [19], to ‘hot, warm and cold’ hydrogen ions [25]. They were also attributed to very high pressures [26] or to spatial distribution variations [12]. In the present experiment, the multicomponent lineshapes are observed only close to the surface of the electrode.

These multicomponent lineshapes might then be attributed to the presence of high electron densities very near the electrodes and lower electron densities in the inter-electrode gap.

Such high electron number densities at the electrodes have already been reported [27–30]. Van der Horst et al [28] studied a nanosecond pulsed discharge in atmospheric pressure H\textsubscript{2}O/N\textsubscript{2} mixtures, with optical emission averaged over a 2 mm gap. They attributed the high electron number densities to a low plasma volume and low plasma on-time, combined with high voltage and current. In recent experiments with NRP discharges in atmospheric pressure air, Minesi et al [29] and Orriere et al [30] observed via fast imaging the presence of 250 μm long filaments with high electron densities near the electrodes. They described these filaments as the precursors of the transition to a thermal spark in the inter-electrode gap.

Recently, Raja et al [31] proposed a new explanation based on numerical simulations of discharges in air. They suggested that n\textsubscript{e} can reach 10\textsuperscript{18} cm\textsuperscript{−3} in the vicinity of the cathode due to secondary electron emission by the high electric field in the thin cathode sheath. A similar phenomenon is likely to occur in water vapor, owing to the high sensitivity of the ionization processes to a variation of the reduced electric field [32]. The high n\textsubscript{e} region would correspond to the region encompassing the thin sheath near the electrodes.

4.2. Electron density in the middle of the gap

To study the gas phase kinetics, we focused on the decay of the electron density at the center of the discharge gap. From the measured data plotted in figure 10, the electron lifetime \( \tau \) (defined as \( n_e(\tau) = n_e(0) / e \)) was determined from an exponential fit of the measurements with \( n_e(0) = 5.0 \times 10^{16} \text{ cm}^{-3} \). We obtained a lifetime \( \tau \) of about 40 ns. Since the \( n_e \) decay curve is almost linear in the semi-log plot, the main path of electron loss is through first-order reactions. Thus, electron-ion recombination reactions are ruled out.

Therefore, we considered the electron attachment reactions
In a previous study, the water dissociation fraction was measured by gas chromatography (GC) of the exhaust of the reactor, and found to be around 4% [39]. The difference of the water dissociation fraction measured in the discharge region (OES, this study) and at the exhaust of the reactor (GC) is likely due to neutral-neutral reactions, such as O(^1D) + H_2O → H_2 + O_2, downstream of the discharge. From these results, we conclude that the main electron loss process is via electron attachment reactions and that 2% of water vapor dissociation is consistent with the previous studies.

5. Conclusions

In summary, we measured the spatial distribution and the temporal evolution of electron number densities in an NRP discharge in water vapor by optical emission spectroscopy to characterize the discharge development and the chemical kinetics. The Stark broadening of atomic lines of H and O showed that the maximum ionization degree in the NRP discharges is up to 20% and 5% in the vicinity of the cathode and of the anode, respectively, and 0.5% in the middle of the discharge gap. The high n_e regions measured near the electrodes suggested the presence of additional electron generation mechanisms driven by the high electric fields in the electrode sheaths. Also, we calculated the lifetime of electrons in the interelectrode gap and found that this result is consistent with a recombination process driven by electron attachment reactions, provided a water dissociation of about 2%.

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