

Article

Spectroscopic Characterization of an Atmospheric Pressure Plasma Jet Used for Cold Plasma Spraying

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Abstract: Cold plasma spray, a powder deposition method by means of an atmospheric pressure plasma jet is a promising coating technology for use on temperature sensitive surfaces. For further improvement of this coating process, a deeper understanding of its thermokinetic properties is required. By means of optical emission spectroscopy, the plasma effluent of an atmospheric pressure nitrogen arc jet is characterized by different distances from the nozzle and different gas flow rates of 35 L min⁻¹ and 45 L min⁻¹. A Boltzmann plot of N₂⁺(B-X) was used to determine rotational temperatures, which were found to be around 4000 K at the nozzle exit. Excitation temperatures, analyzed using atomic nitrogen lines, were around 6000 K for all distances. Stark broadening of the H_α-line was too weak for determination of electron density for both gas flow rates. Overall no influence on gas flow rate was found.

Keywords: cold plasma spraying; atmospheric pressure plasma jet; optical emission spectroscopy; downstream characterization; rotational temperature



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1. Introduction

Thin film deposition has great significance in industrial and medical processes, and numerous studies are concerned with the development of thin-film processes for specific applications [1,2]. Plasma spraying by means of an atmospheric pressure plasma jet (APPJ) allows the deposition of thin layers of metal, metal oxide or polymers and on temperature-sensitive substrates, such as paper or wood. In the process, particles are melted in the plasma of an atmospheric pressure gliding plasma arc jet and applied to the surface [3–5]. The plasma source and parameters ensure low particle temperatures at the nozzle exit compared to a thermal plasma spray, and at working distances of ≥ 20 mm no thermal impact to the specimen occurred. The process is also termed “cold plasma spraying” [6,7].

For broad application, understanding the underlying mechanism and influence of the relevant parameters, process stability and monitoring are necessary. Appropriate methods need to be non-invasive, reliable and fast to detect changes in plasma properties in real time and under process conditions. Optical emission spectroscopy (OES) is well established for the characterization of APPJ because it provides insight into plasma energy distributions and kinetics and is interference-free. OES experiments showed that gliding arcs exhibit an initial quasi-equilibrium zone close to the electrodes, followed by a non-equilibrium zone with electron temperatures about 10 times higher than rotational and translational temperatures [8]. In general, studies showed similar trends for different types of APPJs, including an increase in plasma temperature and electron density with input power or a decrease in plasma temperature with increasing axial distance. Changes in gas composition, well studied for the influence of water, lead to a change in kinetics and therefore a strong variation in plasma temperatures, electron densities, and excited species densities can be observed [9–12]. The presence of substrates can have similar effects, but many jets

are monitored under laboratory conditions, and not many studies focus on substrate interactions [13]. Recent developments have focused on the application of OES for the monitoring and controlling of plasma. Line-ratio methods and simple kinetic models allow evaluation in real time for selected processes [14,15].

To establish OES as a tool to monitor cold plasma spray, fundamental investigations are first necessary. In this study, OES was used for the downstream characterization of the effluent plasma of an APPJ for the thin film deposition of metals, metal oxides or polymers. Beside the determination of reactive species and their development being dependant on the distance from the nozzle and different gas flows, special attention was paid to the determination of plasma temperatures. A rotational temperature, determined by an analysis of the molecular spectrum of a species such as N_2 , N_2^+ , NO or CN, is commonly assumed to be equal to the gas temperature in atmospheric pressure plasmas as long as certain conditions are fulfilled [16,17]. Special care has to be taken in the selection of spectral bands to avoid spectral overlapping of different species. In addition, knowledge of rotational temperature, electron density and electron temperature, also referred to as electron energy, are fundamental. While determining electron density by the Stark effect is possible using OES if the gas temperature is known and the resolution of the spectrometer is sufficient, determining electron energy is sophisticated and requires a valid kinetic model [18,19]. On the other hand, excitation temperatures can easily be derived by line-ratio methods. The general trend for both, electron and excitation temperatures, is the same, while for non-LTE plasmas the electron temperature is higher than the excitation temperature [20,21]. For deposition processes, excitation temperature is of special interest as the energy transfer between excited atoms and molecules is important for the melting of the particles, whereas the energy transfer between electrons and particles is negligible.

2. Materials and Methods

2.1. Plasma Source and Measurement Conditions

The atmospheric-pressure rotating arc jet (Plasma Plotter 3D, INOCON Technologies GmbH, Austria) consisted of a high voltage power supply and a self-developed spray nozzle as an electrode. The plasma was generated as an arc between the high voltage electrode and the grounded spraying nozzle and was blown out by compressed nitrogen (Linde AG, Pullach, Germany, $\geq 99.999\%$) as pictured in Figure 1. The input power of the APPJ was less than 1 kW, described by pulsed high voltage of 3–4 kV (effective voltage 2–3 kV) with a pulse period of 5–10 μ s and a repetition frequency of 50 kHz.

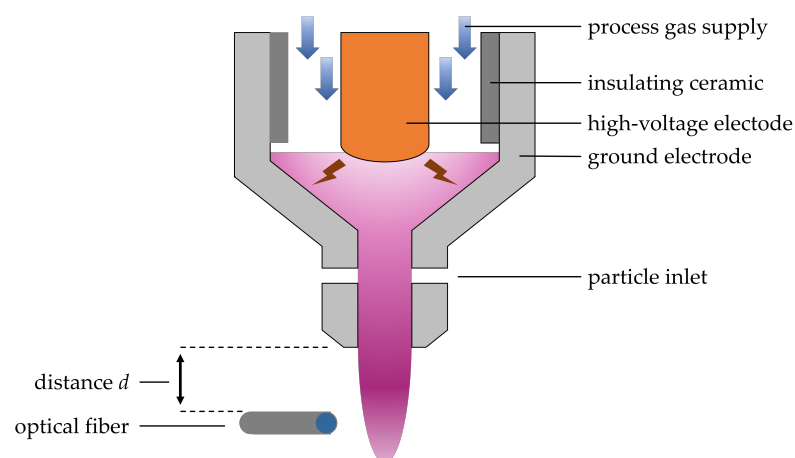


Figure 1. Schematic setup of the atmospheric-pressure plasma jet (APPJ).

Optical emission spectra were taken using an Echelle spectrometer (Aryelle–Butterfly 400, LTB Lasertechnik Berlin GmbH, Berlin, Germany) with two separate spectral ranges—UV (200–355 nm) and UV-VIS (330–960 nm)—with a resolution of ≤ 11 and ≤ 24 pm, respectively, calibrated to wavelength and relative intensity. For spectra recording, a bare optical

fiber (0.22 NA) was placed perpendicular to the plasma torch in distances from 2 to 20 mm relative to the outlet nozzle and at a distance of 3 mm from the plasma column. Thermal impact on the fiber was excluded in prior experiments. Due to the low light intensity of the APPJ, no additional optics or masks were used. For each distance, 10 spectra were taken with an integration time of 50 ms for distances of 2–6 mm and 100 ms for distance of 8–14 mm in the UV region; integration times of 10 ms were taken for distances of 2–10 mm and 100 ms for distances of 12–20 mm in the UV-VIS region. All spectra were dark corrected and averaged for each measurement setting before further evaluation.

2.2. Determination of Plasma Temperatures

Nitrogen and nitrogen ion bands are widely used to determine rotational temperature. Most often, the method is a comparison of the N_2 ($C^3\Pi_u^+ \rightarrow B^3\Pi_g^+$) transition at 337.1 nm with the simulated emission spectra. In our case, the band showed spectral overlap with NO ($B - X$), so it was excluded. The first positive band of N_2 ($B^3\Pi_g^+ \rightarrow A^3\Sigma_u^+$) can also be used for determination, but studies indicate low accuracy for high temperatures [17].

Therefore, the 0-0 band of the N_2^+ ($B^2\Sigma_u^+, v = 0 \rightarrow X^2\Sigma_g^+, v' = 0$) transition at 391.1 nm was chosen to determine rotational temperature as depicted in Figure 2a. Rotational temperature T_{rot} was determined from the slope of the Boltzmann plot, depicted in Figure 2b and given by

$$\ln \left[\frac{I_{nm} \lambda_{nm}^4}{(N' + N'' + 1)} \right] \propto - \frac{hcB_v}{k_B T_{rot}} N' (N' + 1) \quad (1)$$

with the intensity of the transition I_{nm} at the specific wavelength λ_{nm} , quantum numbers of the upper state N' and N'' and h , c , k_B being the Planck constant, speed of light and Boltzmann's constant, respectively. The rotational constant B_v was calculated according to Equation (2) with $B_e = 2.075 \text{ cm}^{-1}$ and $\alpha_e = 0.024 \text{ cm}^{-1}$ taken from [22]:

$$B_v = B_e - \alpha_e(v + 0.5). \quad (2)$$

For the analysis, spectral lines of the P-branch were chosen, and these showed no spectral overlap with lines from the R-branch [17,23]. Rotational lines were identified using values from the literature and are given in Table 1 [24].

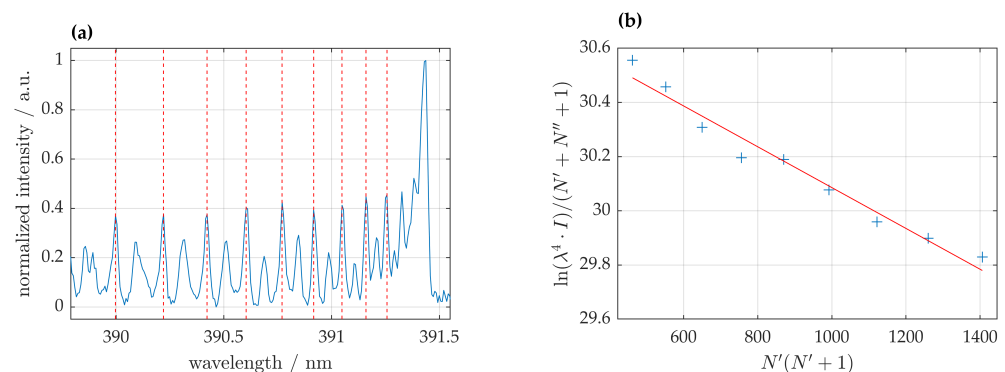


Figure 2. Determination of the rotational temperature of the N_2^+ ($B^2\Sigma_u^+, v = 0 \rightarrow X^2\Sigma_g^+, v' = 0$) transition at a flow rate of 45 L min^{-1} and 2 mm distance from the nozzle. (a) Measured spectra with selected lines. (b) Boltzmann plot.

Table 1. Selected spectral lines for determination of the rotational temperature of the N_2^+ ($B^2\Sigma_u^+, v = 0 \rightarrow X^2\Sigma_g^+, v' = 0$) transition.

N''	λ/nm	$(N' + N'' + 1)$	$N'(N' + 1)$
22	391.17	44	462
24	391.08	48	552
26	391.98	52	650
28	390.85	56	756
30	390.71	60	870
32	390.55	64	992
34	390.37	68	1122
36	390.17	72	1260
38	389.95	76	1406

A set of atomic nitrogen lines was selected to determine the excitation temperature by the Boltzmann plot method and is given by

$$\ln \left[\frac{I_{ki} \lambda_{ki}}{g_k A_{ki}} \right] \propto - \frac{E_k}{k_B T_{\text{exc}}}, \quad (3)$$

where I_{ki} , λ_{ki} and A_{ki} are the spectral line intensity, wavelength and transition probability of the selected transition, respectively, and g_k and E_k are the statistical weight and the energy of the upper level. The selected spectral lines and necessary parameters taken from [25], are listed in Table 2. Uncertainty of A_{ki} is less than $\pm 10\%$ for all transitions.

Table 2. Selected spectral lines for determination of the excitation temperature.

λ/nm	Transition	$A_{ki}(10^6 \text{ s}^{-1})$	E_k/eV	g_k
818.49	$3p^4P^0 - 3s^4P$	8.21	11.844	6
818.80	$3p^4P^0 - 3s^4P$	12.5	11.840	4
859.40	$3p^2P^0 - 3s^2P$	20.9	12.122	2
862.92	$3p^2P^0 - 3s^2P$	26.7	12.126	4
868.03	$3p^4D^0 - 3s^4P$	25.3	11.764	8
868.34	$3p^4D^0 - 3s^4P$	18.8	11.758	6
868.62	$3p^4D^0 - 3s^4P$	11.5	11.753	4
871.17	$3p^4D^0 - 3s^4P$	12.9	11.753	4

2.3. Electron Density

The determination of electron density by Stark broadening is a widely applied method. Often the H_β line is used due to its high sensitivity, but it overlaps the N_2 ($B - A$) band, which is why in this study the H_α line was used. Electron density was calculated by

$$n_e = 10^{17} \times \left(\frac{\Delta\lambda_S^A}{1.098} \right)^{1.47135}, \quad (4)$$

where the electron density is n_e in cm^{-3} , and the Stark broadening is at its full width at half area $\Delta\lambda_S^A$ in nm. A deconvolution of the measured line profile and the determination of $\Delta\lambda_S^A$ was performed as described in [26–28]. In this experiment, nitrogen with the addition of 1% hydrogen (Linde AG, Pullach, Germany, $\geq 99.999\%$) was used as a working gas, and measurements were taken at a distance of 2 mm from the nozzle.

3. Results

Besides emission from the OH, N_2 and N_2^+ molecular bands and from atomic nitrogen and atomic oxygen in the spectra, traces of iron and tungsten were identified. While the metallic species are due to erosion of the electrode, the other species originated from the feed gas and the surrounding air.

For both spectral ranges, the emission intensity decreased exponentially. To compare results from both spectral ranges, all spectra were normalized to the intensity of N_2 ($C - B$) at 337.1 nm, which was observed in both.

Close to the nozzle in the UV-region from 200 to 355 nm the second positive system (SPS) of molecular N_2 ($C^3\Pi_u^+ \rightarrow B^3\Pi_g^+$) was the dominant species, and OH ($A - X$), NO ($A - X$) and NO ($B - X$) were present. With increasing distance to the nozzle N_2 emission intensity decreased while NO and OH intensities increased with a maximum intensity relative to N_2 at a distance of 10 mm from the nozzle as depicted in Figures 3 and 4.

In the UV-VIS region from 330 to 960 nm different nitrogen species were present. The first negative system (FNS) of N_2^+ ($B^2\Sigma_g^+ \rightarrow X^2\Sigma_g^+$) was clearly visible and of higher intensity than the N_2 ($C - B$) transition. For distances of 2 and 14 mm the 0-0 transition of the FNS showed no spectral overlap with other species and was thus suitable for rotational temperature determination.

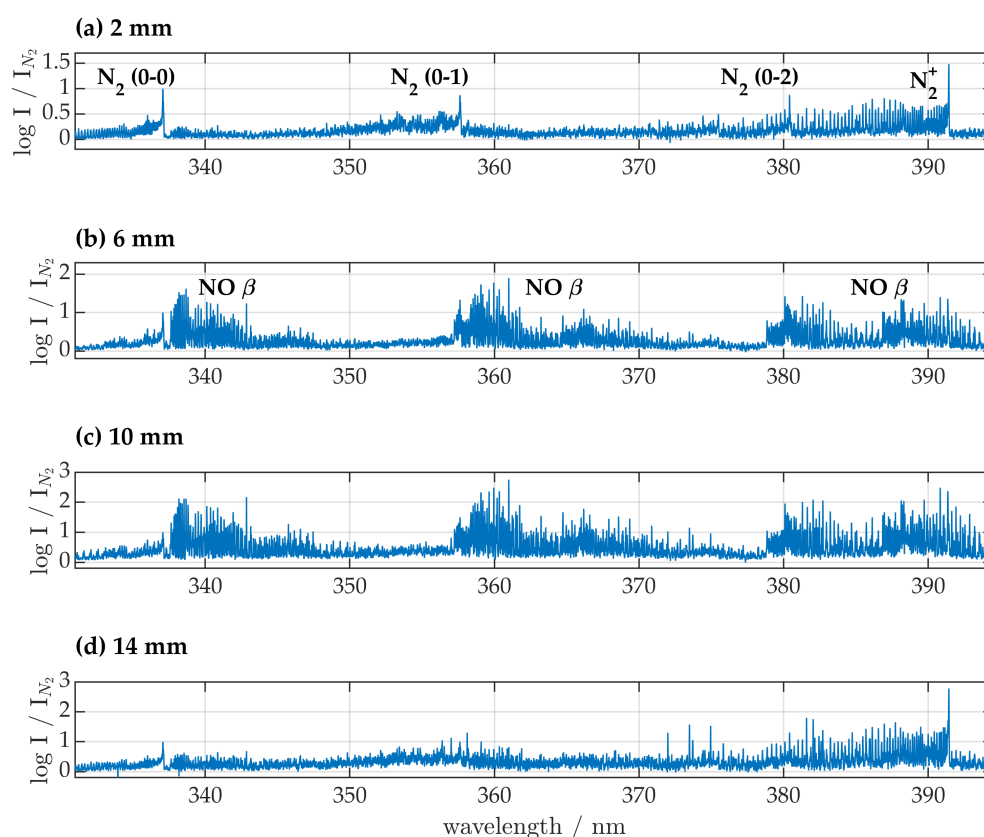


Figure 3. Spectra in the wavelength range of 333.1–394.5 nm at a gas flow of 35 L min^{-1} . For (a) 2 mm, (b) 6 mm, (c) 10 mm and (d) 14 mm distance to the nozzle.

The first positive system of N_2 ($B^3\Pi_g^+ \rightarrow A^3\Sigma_u^+$) led to characteristic spectral features for wavelengths above 500 nm. Intensities of atomic nitrogen species were very high for all distances. Atomic oxygen ($3p^5P \rightarrow 3s^5S$) at 777 nm was clearly visible, but no other oxygen transitions were detected. With increasing distance to the nozzle an increase in FNS, FPS, atomic nitrogen and atomic oxygen relative to SPS was detected. No significant differences were found for gas flows of 35 L min^{-1} and 45 L min^{-1} .

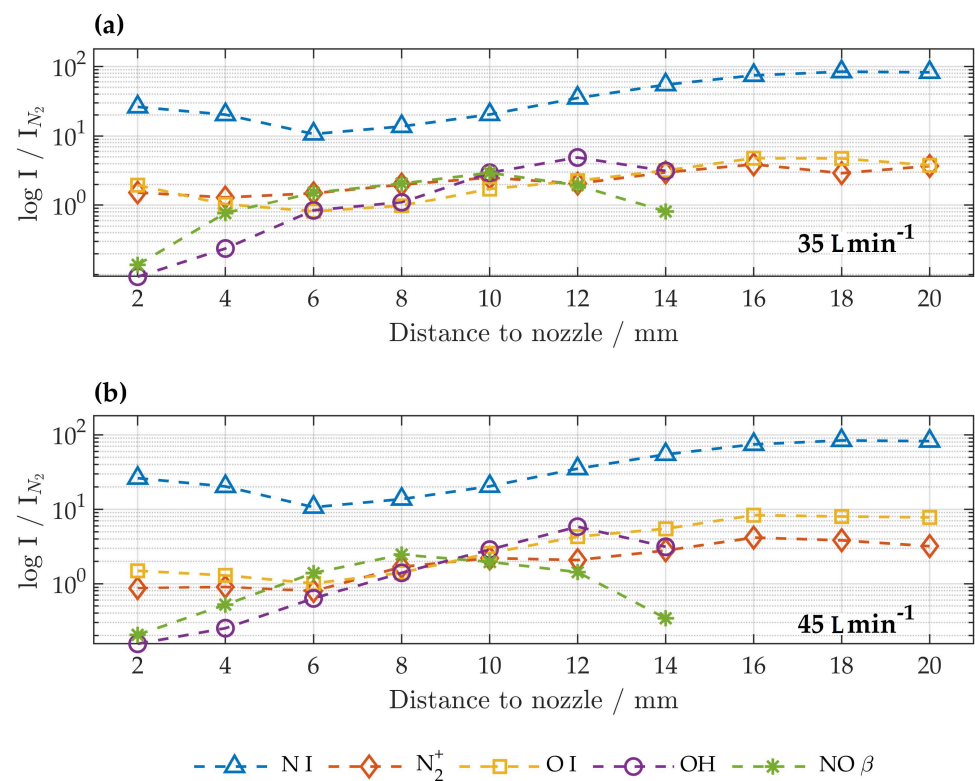


Figure 4. Intensity of different species normalized to N_2 intensity. For (a) 35 L min^{-1} gas flow and (b) 45 L min^{-1} gas flow.

3.1. Rotational and Excitation Temperatures

Rotational and excitation temperatures were determined according to Equations (1) and (3) for all spectra. The error was determined by propagation of the slope error. Only results with an correlation coefficient >0.98 and errors below 15% were considered for further evaluation and are presented in Figure 5.

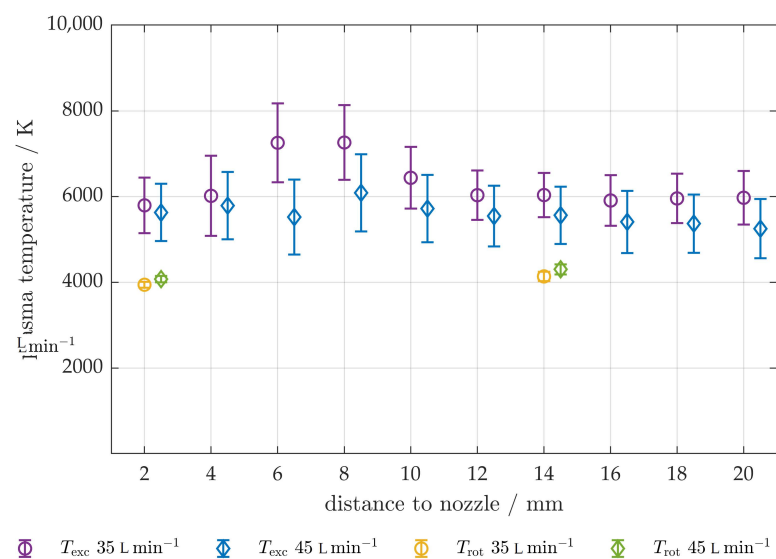


Figure 5. Rotational temperature and excitation temperature for gas flows of 35 L min^{-1} and 45 L min^{-1} and different distances from the nozzle. For a gas flow of 45 L min^{-1} the x-axis was shifted for better visibility.

Rotational temperatures could only be determined for distances of 2 and 14 mm from the nozzle; for all other distances the correlation coefficient was <0.98 due to spectral overlap with other species. For a gas flow of 35 L min^{-1} rotational temperatures were found to be $3940 \pm 70 \text{ K}$ at a distance of 2 mm and $4100 \pm 100 \text{ K}$ at 14 mm. Rotational temperatures for 45 L min^{-1} were slightly higher with $4070 \pm 70 \text{ K}$ at a distance of 2 mm and $4300 \pm 110 \text{ K}$ at 14 mm.

Excitation temperatures show a mean value of 6247 K for lower and 5575 K for higher gas flow. Comparison of the determined values shows no influence on the distance to the nozzle or on different feed gas flows of 35 L min^{-1} and 45 L min^{-1} .

All values are depicted in Figure 5.

3.2. Electron Density

Electron densities were determined assuming that the N_2^+ rotational temperature corresponded to the gas temperature. No influence of self-absorption was detected in the spectra under experimental conditions. Determined values were below the recommended limits for Equation (4) of $5 \times 10^{14} \text{ cm}^{-3}$ for both gas flows [27,29].

4. Discussion

For the APPJ under investigation, no difference in emitted species and plasma parameters were found for different gas flows of 35 L min^{-1} and 45 L min^{-1} . Similar results are in the literature, where properties of APPJ are independent of gas flow rates over a wide range [10,17].

As depicted in Figure 5, excitation temperatures are about 1.5 times higher than rotational temperatures; hence, it can be assumed that the APPJ is in a non-equilibrium state. Both the rotational and excitation temperatures of the nitrogen species were found to be high at the nozzle exit and steady with distance, while their intensity and concentration decreased. Although the measurement series was consistent, some uncertainties needed to be addressed. It is known, that $\text{N}_2^+(\text{B})$ molecules are not only populated by direct excitation from the ground state but are also by highly vibrationally excited $\text{N}_2(\text{X})$ molecules through resonant energy transfer [30]. These “hot” molecules cannot be detected by simple Boltzmann plots; therefore, results should be confirmed by rotational temperature determination of further bands or systems or by the application of other methods.

For most plasma jets, gas temperatures were reported to decrease with increasing axial distance. This behaviour was due to convective cooling and the entrainment of ambient air as shown by Hsu et al., who studied the effect of ambient air on the gas temperature of a nitrogen APPJ using a glass tube for shielding and thermocouples for measuring [11]. Xiao et al. reported on a nitrogen APPJ, where excitation and rotational temperatures measured by OES were not dependent on distance [10]. For long axial distances the approximation of the rotational temperature equalling the gas temperature seemed not to be valid. While for long distances, the gas temperature was mainly governed by cold neutral temperatures from the ambient air, which still had a fair amount of reactive plasma species, such as N_2^+ and atomic nitrogen with high rotational and excitation temperatures.

Former measurements on APPJs for cold plasma spray using thermocouples showed low gas temperatures of $\leq 400 \text{ K}$ at a working distance of 20 mm from the nozzle exit [5,7]. While differences between gas and rotational temperatures were one explanation, it should be noted that in our experiment no particles for deposition were added to the jet, and studies showed a great influence of substrates on gas-phase chemistry [13]. As the validity range for electron-density determination was exceeded, it can only be stated that the electron density was lower than the limit of $5 \times 10^{15} \text{ cm}^{-3}$ although this is in agreement with the literature for atmospheric-pressure non-thermal nitrogen jets [31].

5. Conclusions

In this study, a gliding arc atmospheric pressure plasma jet for thin-film deposition by cold plasma spray was examined using optical emission spectroscopy. Both the excitation

and rotational temperatures seemed to be independent of distance. If previous studies were taken into account, where a similar gliding arc APPJ showed low particle temperatures and no thermal impact on the substrate, the rotational temperatures seemed to be high with values of 3940 ± 70 K and 4070 ± 70 K for the different gas flows. It was assumed that the addition of particles may have had a cooling effect. Therefore the impact of the addition of metal particles shall be addressed in further investigations. In addition, numerical gas-flow simulations will be carried out, to take into account the special geometry of the nozzle and the current data on excited species and plasma temperatures.

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