Investigation on Optical Properties of Atmospheric Pressure Plasma Jets of N$_2$ Gas

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Abstract

In this study, firstly, N$_2$ atmospheric pressure plasma jet (APPJ) system was presented. Nitrogen gas discharges are produced as jet using an AC power supply which can be adjusted between 6-18 kV and the frequency value of 13-20 kHz at atmospheric pressure. The change of length of produced atmospheric pressure nitrogen plasma jet, according to gas flow rate has been investigated and the produced jet length was approximately 2 cm for 5 L/min when the applied voltage was 18 kV and the frequency was 15 kHz. Nitrogen plasma jet produced at atmospheric pressure was examined with optical emission spectroscopy (OES) and the correlation between gas flow rate and emission spectra were investigated. Furthermore, electron temperature and electron density of atmospheric pressure nitrogen gas plasma jet were estimated under different flow rates of N$_2$ gas.

Keywords: Nitrogen; Atmospheric pressure plasma; N$_2$ APPJ; Electron temperature; Electron density.
N₂ Gazi Atmosferik Basınç Plazma Jetlerinin Optik Özelliklerinin İncelenmesi

Öz

Bu çalışmada öncelikle atmosferik basınçta plazma jet (APPJ) üretimine olanak sağlayan sistem tanıtılmıştır. Azot gazı deşarjları atmosferik basınçta 6-18 kV ve 13-20 kHz ayarlı AC güç kaynağı ile jet olarak üretilmiştir. Üretilen atmosferik basınç azot plazma jetin gaz akış hızına göre uzunluğunun değişimi incelenmiş olup, üretilen jet uzunluğu 5 L/dk gaz akış hızı, 18 kV voltaj ve 15 kHz frekans değerinde yaklaşık olarak 2 cm’dir. Atmosferik basınçta üretilen azot plazma jet, optik emisyon spektroskopisi (OES) ile incelenmiş ve gaz akış hızı ile emisyon spektrumlarındaki değişimler belirlenmiştir. Bununla birlikte, atmosferik basınç azot gazi plazma jetin elektron sıcaklığı ve elektron yoğunluğu azot gazının farklı gaz akış hızları için hesaplanmıştır.

Anahtar Kelimeler: Azot; Atmosferik basınç plazma; N₂ APPJ; Elektron sıcaklığı; Elektron yoğunluğu.

1. Introduction

Many studies have been carried out so far with gas discharge plasmas known as cold plasmas. Cold plasmas have many advantages such as the effects of low temperature, low electric field, and the chemical interactions of the active radicals have various applications. Cold plasmas are widely used in many applications such as sterilization, surface applications such as coating, activation, cleaning, polymerization, oxidation, nitriding, and medical treatments [1-4]. Instead of noble gases such as argon (Ar), helium (He), etc., nitrogen (N₂) gas has also used in these applications [5, 6]. Nitrogen gas plasmas have a vital importance because of the content of radical (reactive) and metastable particles. Molecular nitrogen does not react easily under normal conditions and is ineffective. However, the excited or dissociated N₂ species, especially atomic nitrogen (N) in the mixed gases containing N₂ or N₂⁺ caused many reactions to be used in important applications. Different excited states of species such as N₂, N₂⁺ and N are also formed in nitrogen plasmas and form important reactions [7]. The first excited state of nitrogen, the metastable triplet N₂(A³Σ_u⁺), has a threshold energy of 6.2 eV and a lifetime of about 2 seconds. From this point of view, it is an important energy carrier, so it plays an effective role in N₂ plasma by making important mechanisms such as ionization, decomposition, plasma chemistry and gas heating [8]. Although N₂(A) plays an important role in the basic processes controlling nitrogen discharge, it is known that the main energy storage is not by electronic metastable. It has been observed in previous studies that the N₂(X¹Σ_g⁺) state of the control mechanism is at vibration levels [9]. However, N atoms formed by the decomposition of nitrogen molecules used in metallic
nitriding are easily produced in nitrogen plasma. Observation of \( N_2^+(B^2\Sigma_u^+\rightarrow X^2\Sigma_g^+) \) transitions also indicates that \( N_2^+ \) is abundant [10]. Characteristic emissions and numerical calculations of species such as \( N_2, N_2^+, N^+ \), and \( N \) in low- and atmospheric pressure of nitrogen luminescent (glow) discharges have been examined [11, 12]. \( N_2 \) and \( N_2^+ \) species in nitrogen plasma play vital role in synthesis of new functional and mechanical materials as they have strong chemical activity. Production of anti-microbial low density polyethylene films and hydrophilic polymer structures [13], processing of hydroxyl cellulose films [14], GaN nanostructures containing nano-wire and nano-particles [15], nitrogen oxide processing [16], production of high activity plasma welding for the storage of silicon nitride films [17], modification of amorphous \( \text{SiO}_2 \) nanoparticles [18], single crystal production [19], modification of stainless steel surfaces (ion implantation) [20], treatment of indium tin oxide (ITO) films [21], graphene and graphite structures [22], surface modification of polyacrylonitrile copolymer structures [23], diamond building carbon (DLC) production [24], annealing \( \text{Ta}_2\text{O}_5 \) films [25] are examples of applications of material processing of cold plasma produced with low pressure nitrogen gas/gas mixtures.

According to the literature, there are a lot of applications by using \( \text{N}_2 \) APPJs, but the basic characteristics of the produced plasma are not examined in detail. In order to do that, a jet system which produces nitrogen gas plasmas at atmospheric pressure is designed and produced. Firstly, the behavior of nitrogen gas plasma in atmosphere medium according to different gas flow rate was investigated. Then, optical emission spectra (OES) of nitrogen plasmas (\( \text{N}_2 \) APPJs) were taken depending on gas flow rate. The possible atomic and molecular transitions in nitrogen plasma were obtained from the OES data in accordance with the literature. Finally, electron temperature \( (T_e) \) and electron density \( (n_e) \), which are one of the main parameters of nitrogen gas plasma, were calculated by Boltzmann two-line method. It is considered that this study would be a basis for determining the appropriate gas flow rate for \( \text{N}_2 \) plasmas in further experimental studies.

2. Materials and Methods

Designed system to produce \( \text{N}_2 \) APPJs and the scheme of \( \text{N}_2 \) APPJ system were given in Fig. 1 and Fig. 2, respectively. The atmospheric pressure plasma system used here is known as plasma jet.
The whole system was installed in a fume hood to protect against the stifling effects of nitrogen gas. In order to generate APPJ, three identical quartz glass pipes (OD: 6 mm, ID: 4 mm) were used. First pipe which was used as gas inlet was fixed on rectangle shaped plexiglass (50x25x37 mm). Other pipe which was fixed on the plexiglass was used to hold tungsten electrode. The tungsten electrode was extended approximately 3 mm to the nozzle of the pipe attached to gas outlet. The tungsten wire has 0.5 mm thickness. The distance of the ring electrode to the nozzle of the pipe was determined as a result of making a series of attempts. A ring electrode which was made of copper was connected to the end of the glass pipe. Then, a gas flowmeter (LZT M-6 flowmeter, 2-10 L/min) was connected to control gas flow rate to gas inlet of the system. Gas outlet of the pipe was mounted with pneumatic hose (OD: 10 mm, ID: 6 mm).
Nitrogen gas cylinder (Habas 99.999% purity) and its regulator were used to supply gas to the APPJ system. The connection between this gas cylinder and the fume hood is provided by a 6.5 mm diameter pneumatic hose. The alternating current (AC) power supply (ELES HV-711GK4), which can be adjusted between 6-18 kV and the frequency value of 13-20 kHz, was used for generating N<sub>2</sub> APPJ.

A spectrometer (Ocean Optics USB2000+), fiber optic cable and computer program (OceanView software) were used in order to examine the spectroscopic properties of N<sub>2</sub> APPJ as can be seen in Fig. 1. 10 mm wide slit (300 grooves/mm grating, the spectral resolution = 0.1 nm) were used to measure spectra in the sensitive range from 200-1100 nm. In order to avoid fluctuation of intensity, integration time of OES was fixed at 1 s through the experiment, and optical emission spectrometer was recalibrated priorly each measurement.

3. Results

In order to generate N<sub>2</sub> APPJ, the high voltage input was connected to the tungsten needle electrode. A ring electrode which was made of copper was connected to the end of the glass pipe. Then, N<sub>2</sub> gas was flowed through the glass pipe in the middle of the plexiglass. Gas sent from the nitrogen gas cylinder was read out from gas flowmeter, simultaneously. After all, when the AC voltage was applied between the electrodes, the nitrogen discharge was generated between the electrodes. When the flow rate of nitrogen gas was set at a certain value, the nitrogen gas discharge between the electrodes emerges as a N<sub>2</sub> APPJ. At different gas flow rates, generated N<sub>2</sub> APPJ was shown in Fig. 3.

![Figure 3: Atmospheric pressure nitrogen gas plasma jet photographs for different N<sub>2</sub> flow rates](image-url)
We have achieved the longest jet length (about 2 cm) at 18 kV-15 kHz in N₂ APPJs that we produced in a similar diameter before. When we reduce the voltage to less than 18 kV, there is a decreasing in the intensity of the jet, thus its length. On the other hand, if we increase the frequency above 15 kHz, it turned to more intense plasma but similarly jet length decreased. Moreover, when the frequency value was adjusted below 15 kHz, the jet becomes unstable and discontinuous form. The distance between the ring electrode and the nozzle was set at 3 cm. Proper adjustment of this distance prior to the experiment affects the structure of the plasma formed in the atmosphere. It was seen that when the distance between the electrodes decreased, the plasma transformed into an arc form with a high intensity, in other respects when it increased, plasma formed only between the electrodes and could not reach the atmosphere.

The change of plasma jet length that can be released into the atmosphere according to the gas flow rate was also shown in the Fig. 4.

![Figure 4: The change of plasma jet length according to the gas flow rate](image)

As shown in Fig. 3 and Fig. 4, the length of APPJ was proportional to gas flow rate. This situation was related with high pressure formed inside the glass pipe. It was observed that high pressure nitrogen gas mixed with the atmosphere medium had been seen to ionize more easily. Spectra taken from the same distance of the N₂ APPJs were shown in Fig. 5.
In the optical emission spectra taken for different nitrogen gas flow rates, various atom/atoms, molecules and radical particles were found in the N$_2$ APPJs [26]. Here, it was observed that the NO radical concentration was proportional to gas flow rate. Stated in other words, the NO radical concentration increases as gas flow rate increases. OH radicals (at 308 nm) were dominated by the NO radicals. Therefore, the wavelength corresponding to the OH radicals in the optical emission spectra was not marked and was only given in Table 1. The intensity of N atoms (747 - 870 nm) was determined to be quite low compared to other species such as N$_2$, N$_2^+$, etc. On the other hand, N$_2$ and N$_2^+$ peaks were observed to increase continuously up to 5 L/min (Fig. 6). As a result of ionization of nitrogen gas, this is an expected result and it is clearly seen from the Fig. 6 that ionization increases with increasing gas flow rate.
Furthermore, OES device used during the measurement reached the upper count limit at a flow rate of 5 L/min. When working in atmosphere medium, wavelengths of H, N and O are expected to be seen in the spectra. However, since the OH and NO radicals were formed by the interaction of H, N, and O atoms, these atoms were not directly observed in the spectra. OH and NO intensities were supposed to be suppressed by N\textsubscript{2}. Observed transitions in N\textsubscript{2} APPJs were given in Table 1.

Table 1: Observed atomic and molecular transitions in N\textsubscript{2} APPJ [26]

| Plasma Component | Wavelength (nm) | Transition | Excitation Energy (eV) |
|------------------|----------------|------------|------------------------|
| NO               |                |            |                        |
|                  | 204.70         | ΛΣ\textsuperscript{+}, (v=2) – Σ\textsubscript{2} (v=0) | -5.46 |
|                  | 214.80         | ΛΣ\textsuperscript{+}, (v=1) – Σ\textsubscript{1} (v=0) | -5.46 |
|                  | 226.20         | ΛΣ\textsuperscript{+}, (v=0) – Σ\textsubscript{1} (v=0) | -5.46 |
|                  | 236.30         | ΛΣ\textsuperscript{+}, (v=0) – Σ\textsubscript{1} (v=1) | -5.46 |
|                  | 247.10         | ΛΣ\textsuperscript{+}, (v=0) – Σ\textsubscript{1} (v=2) | -5.46 |
|                  | 258.70         | ΛΣ\textsuperscript{+}, (v=0) – Σ\textsubscript{1} (v=3) | -5.46 |
|                  | 271.30         | ΛΣ\textsuperscript{+}, (v=0) – Σ\textsubscript{1} (v=4) | -5.46 |
| OH               | 308.00         | ΣΣ\textsuperscript{−} (v=0), – ΣΣ\textsuperscript{+} (v=0) | 9.10 |
|                  | 315.90         | ΣΠ\textsuperscript{0}, (v=1) – ΣΠ\textsuperscript{0}, (v=0) | 11.30 |
|                  | 337.10         | ΣΠ\textsuperscript{0}, (v=0) – ΣΠ\textsuperscript{0}, (v=0) | 11.00 |
|                  | 353.60         | ΣΠ\textsuperscript{0}, (v=1) – ΣΠ\textsuperscript{0}, (v=2) | 11.30 |
|                  | 357.70         | ΣΠ\textsuperscript{0}, (v=0) – ΣΠ\textsuperscript{0}, (v=1) | 11.00 |
|                  | 370.90         | ΣΠ\textsuperscript{0}, (v=2) – ΣΠ\textsuperscript{0}, (v=4) | 11.50 |
|                  | 375.40         | ΣΠ\textsuperscript{0}, (v=1) – ΣΠ\textsuperscript{0}, (v=3) | 11.30 |
|                  | 380.40         | ΣΠ\textsuperscript{0}, (v=0) – ΣΠ\textsuperscript{0}, (v=2) | 11.00 |
|                  | 399.70         | ΣΠ\textsuperscript{0}, (v=1) – ΣΠ\textsuperscript{0}, (v=4) | 11.30 |
|                  | 405.80         | ΣΠ\textsuperscript{0}, (v=0) – ΣΠ\textsuperscript{0}, (v=3) | 11.00 |
|                  | 434.30         | ΣΠ\textsuperscript{0}, (v=0) – ΣΠ\textsuperscript{0}, (v=4) | 11.00 |
| N\textsuperscript{2}+ | 391.40       | ΣΣ\textsuperscript{−}, (v=0) – ΣΣ\textsuperscript{−}, (v=0) | 18.70 |
|                  | 427.80         | ΣΣ\textsuperscript{−}, (v=0) – ΣΣ\textsuperscript{−}, (v=1) | 18.70 |
|                  | 470.90         | ΣΣ\textsuperscript{−}, (v=0) – ΣΣ\textsuperscript{−}, (v=2) | 18.70 |
| N                 | 746.80         | 3\textsuperscript{p} – 3\textsuperscript{p}\textsuperscript{4}\textsuperscript{S} | 11.90 |
|                  | 870.30         | 3\textsuperscript{p} – 3\textsuperscript{p}\textsuperscript{4}\textsuperscript{D} | 11.80 |
In the non-LTE plasmas, the electron temperature \( T_e \) can be calculated using Boltzmann approximation [27].

\[
T_e = \frac{E_2 - E_1}{k} \left[ \ln \left( \frac{A_2 g_2 I_1 \lambda_1}{A_1 g_1 I_2 \lambda_2} \right) \right]^{-1}
\]  

(1)

Here, sub index 1 and 2 correspond to two different electronic states of N\(_2\). \( E_1 \) and \( E_2 \) represent the energy levels. \( \lambda_1 \) and \( \lambda_2 \) are wavelengths of emitted photons; \( I_1 \) and \( I_2 \) are measured relative intensities. \( g_1 \) and \( g_2 \) represent the statistical weights of these levels. \( A_1 \) and \( A_2 \) represent the transition probabilities. Furthermore, the electron density can be estimated as follows [28]:

\[
n_e \equiv 10^{18} T^{7/2} \left( \frac{1}{n_a} \right)^2 \left( \frac{2}{n_b} \right)^5 \text{ cm}^{-3}
\]  

(2)

In Eqn. (2), \( n_a \) and \( n_b \) are the level of excited state and ground state, respectively. \( T \) is the electron temperature. Electron temperature of N\(_2\) APPJ was estimated with using N atoms (at 747 nm and 870 nm) and the values of \( T_e \) varied from 0.19 eV to 0.31 eV for different gas flow rates as can be seen in Fig. 7. The electron densities for different gas flow rates were also calculated as can be seen in Fig. 8. The measurement results obtained in the experiment for this calculation are given in Table 2.

### Table 2: Data used in electron temperature calculation

| Flow Rate | Wavelength (nm) | Intensity (Arb.u.) | \( A_1 g_1 \) (s\(^{-1}\)) | \( A_2 g_2 \) (s\(^{-1}\)) | \( E_1 \) (eV) | \( E_2 \) (eV) |
|-----------|-----------------|--------------------|-----------------------------|-----------------------------|--------------|--------------|
| 2 L/min   | 746             | 379                | 7.84 \times 10\(^7\)       | -                           | 11.9         | -            |
|           | 870             | 362                | 4.32 \times 10\(^7\)       | -                           | 11.75        |
| 3 L/min   | 746             | 377                | 7.84 \times 10\(^7\)       | -                           | 11.9         | -            |
|           | 870             | 300                | 4.32 \times 10\(^7\)       | -                           | 11.75        |
| 4 L/min   | 746             | 376                | 7.84 \times 10\(^7\)       | 11.9                        | -            |
|           | 870             | 393                | 4.32 \times 10\(^7\)       | -                           | 11.75        |
| 5 L/min   | 746             | 434                | 7.84 \times 10\(^7\)       | 11.9                        | -            |
|           | 870             | 332                | 4.32 \times 10\(^7\)       | -                           | 11.75        |
As N₂ gas flow rate increased, there was no linear change in electron temperature. It is assumed that this result is due to the production of N₂ APPJ in the fume hood. At high gas flow rates, the jet draws towards the top of the furnace and leans towards the nearest electrode. Here, the outgoing plasma jet may tend to make a new ionization line. Since this ionization line can generate new types of reactions, it is thought that such variations are occurred in temperature.
calculations. Similar to the change of electron temperature, as \( \text{N}_2 \) gas flow rate increased, there was no linear change in electron temperature. The electron densities obtained from the \( \text{N}_2 \) APPJ were between \( 1.14 \times 10^{18} \text{ cm}^{-3} \) and \( 1.87 \times 10^{18} \text{ cm}^{-3} \) values. It is thought that the reason for the decrease of electron density at high gas flow rates (especially at 4 L/min) is due to the ionization of gas atoms by free electrons. Similar results were obtained as the experiments were repeated. Calculated \( T_e \) and \( n_e \) values in accordance with the literature [27].

4. Conclusions

In this study, atmospheric pressure nitrogen gas plasmas (\( \text{N}_2 \) APPJs) were produced as a jet. The experiments were carried out in fume hood in order to protect from the effects of harmful gases which may occur in \( \text{N}_2 \) APPJs produced in atmospheric medium. The production of nitrogen gas plasma jet is quite difficult in comparison to noble gases. However, we have successfully produced plasma jet longer than 2 cm by using the alternating current power supply. The length of the jet was closely related to gas flow rate. It was determined that the jet length increased in proportion to the increase in gas flow rate. At high gas flow rates, APPJ bend towards the nearest electrode. The longest jet was observed at a gas flow rate of 4-5 L/min. However, therefore bending has also occurred at these flow rates. As the connection part of the ring electrode to the power supply in the system was close to the nozzle, it has been observed that the ionization line was directed towards this electrode as the length of the jet increases. The distance of the ring electrode to the end of the pipe was determined by trial and error. Plasma arc formation was observed if the ring electrode was close to the nozzle of the tube, and jet formation was not observed if the ring electrode was away from the nozzle of the tube. After that, the optical emission spectra were taken from 0.5 cm distance of the \( \text{N}_2 \) APPJ system. In the obtained spectra, as gas flow rate increased, \( \text{N}_2, \text{N}_2^+ \), and NO peaks increased proportionally. In addition, the OH peaks increased depending on gas flow rate, but these peaks were dominated by NO. Therefore, the wavelength corresponding to the OH radicals in the optical emission spectra was not marked and was only given in Table 1. Furthermore, electron temperature and electron density were calculated according to gas flow rate by using spectral line intensities of N atoms. Although the nitrogen gas flow rate increased, there was a relatively small increase in electron temperature, but a linear increase was not achieved. At the same time, the outgoing jet was trying to make a new ionization line. This is thought to increase the electron temperature and hence the electron density. Detailed studies on the electrical properties of \( \text{N}_2 \) plasma jet applications, in particular voltage and frequency changes, are planned for the future.

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