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Osaka University
Fundamental Characteristics of a Microwave Discharge Type
Plasma Source Working under Atmosphere Pressure†

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Abstract

The microwave discharge plasma generated at low pressure has been used in many industrial productions as a manufacturing method for etching, or deposition, because it is clean and has high chemical reactivity. Recently, studies for microwave discharge plasma at atmospheric pressure also have begun. It has the advantage of low cost, because the costly vacuum chamber and pump for evacuation are not required for plasma generation. As the atmospheric microwave discharge plasma is also clean and has high chemical reactivity, it is expected to be used for the resolution and detoxification of environmental pollution gas and for surface treatment and electromagnetic coating at atmospheric pressure. In this work, the atmospheric microwave discharge plasma source has been developed and its fundamental characteristics investigated. Mixtures of argon and nitrogen gas were used for the working gases with a 2.45GHz microwave up to 600W. In the microwave power range, Ar was only discharged by adding N₂. This is similar to the Penning effect. Radiant intensity of the atmospheric microwave discharge plasma increased with an increase of N₂ in the working gas. By spectroscopic analysis, it was found that the emission was mainly produced by excitation of N₂ particles.

KEY WORDS: (Atmospheric Microwave Discharge Plasma Source), (Emission Spectroscopy), (Microwave Discharge), (Atmospheric Pressure), (Electric Properties)

1. Introduction

Plasma technologies have been widely applied to various fields such as electrical and electronic engineering, development of new materials, preservation of environment, and space propulsion. Rapid progress of the technology produced a plasma display, VLSI, multifunctional materials and provided a method of resolution of environmental pollution gases.

Low pressure plasma processing is popularly used for surface modification and gas treatment because of the stability of the discharge. A vacuum chamber is however necessary for low pressure plasma generation and is costly and needs large amounts of electricity for He evacuation. On the other hand, in atmospheric pressure plasma processing, the costly vacuum chamber is not required. So, stable atmospheric pressure plasma generation is expected to be applicable in various industries.

One of the authors had already developed a high power plasma jet, was called a gas tunnel type plasma jet, and its performance was described in previous studies. For example, the plasma jet produced by a 200kW Class gas tunnel type plasma jet produces a high temperature of more than 20,000K, high energy density, and high thermal density of 80%. These properties are superior to those of other conventional type plasma jets. Therefore this plasma has a great potential for various applications in thermal processing; high quality ceramic coatings by the gas tunnel type plasma spraying method, and alumina coatings with Vickers hardness of Hv=1200-1600. As another application, the gas tunnel type plasma jet was applied to the surface nitridation of titanium. Also, the speedy formation of a high functional thick TiN coating was investigated.

In general, an atmospheric pressure plasma generator with DC discharge consumes considerable amounts of electric power and its electrodes are severely damaged because of the high temperature. These limit the application field of the atmospheric pressure plasma generator. However, for applications such as surface modification and gas treatment, high temperature is not always necessary. For various applications, a microwave discharge type plasma source working under atmosphere pressure (atmospheric microwave discharge plasma source) has also been developed in our laboratory. This plasma source utilizes no electrode for plasma production, so the plasma source will assure long life time and will ensure reliable operation because it avoids the...
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performance decrement due to the degradation of the electrodes. The plasma source can keep a stable discharge under atmospheric pressure after ignition by using a thin tungsten wire and can be operated with about 600W of electric power.

The microwave discharge plasma generated at low pressure has been used in many industrial productions such as semiconductor and optical component production as a device for etching or deposition because it is clean and has high chemical reactivity. Studies of microwave discharge plasma at atmospheric pressure have also begun recently. As the atmospheric microwave discharge plasma has also these advanced characteristics, it is expected to be used for the resolution and detoxification of environmental pollution gases such as chlorofluorocarbon and nitrogen oxides from industrial facilities and cars and for the surface treatment and the electromagnetic coating at atmospheric pressure.

In this work, an atmospheric microwave discharge plasma source has been fabricated and its fundamental characteristics investigated. Premixed gas of argon (Ar) and nitrogen (N₂) was used as the working gas with a 2.45GHz microwave up to 600W. Electrical characteristics were measured under various mixture and power conditions. The constituent elements of the atmospheric microwave discharge plasma were examined using the emission spectroscopy. The state of atmospheric microwave discharge was observed with a high-speed framing camera.

2. Experimental Description
2.1 Atmospheric microwave discharge plasma source
The atmospheric microwave discharge plasma source is schematically shown in Fig. 1 and its photograph is in Fig. 2. The plasma source comprises a 2.45GHz microwave power supply (maximum power of 600W), a plasma torch of a glass tube, a reflectionless termination, and a gas supply system. Microwaves are introduced into the plasma torch through the tree stub tuner and the power monitor. A premixed gas of Ar and N₂ gas is produced by using the gas supply system and is introduced into the plasma torch. Then, atmospheric plasma is ignited in the plasma torch when a thin tungsten wire is used for discharge. In the experiments with the atmospheric plasma generation, the three stub tuner is frequently adjusted to minimize reflected microwave power.

2.2 Fundamental characteristic measurements of the atmospheric microwave discharge plasma source
An atmospheric microwave discharge was not ignited with a working gas of only Ar. By adding a little amount of N₂ to the working gas, the discharge occurs. This is similar to the Penning effect, because the discharge was not also ignited with only N₂ as a working gas. However, the N₂ ionization voltage of 15.58eV is larger than the metastable state for Ar of 11.53 and 11.72eV. The stably operating range of the plasma source was investigated under several experimental conditions listed in Table 1.

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<th>Microwave Power (w)</th>
<th>Working gas flow rate (l/min)</th>
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<td>0 ~ 600</td>
<td>Ar 4 ~ 13   N₂ 0.2 ~ 3</td>
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Microwave absorption factors: ηₚ at several N₂ concentrations in working were measured. To maintain the atmospheric pressure discharge, the N₂ concentration was changed. The microwave absorption factor was defined here as follows.

\[ η_p = \frac{P_{in} - P_{loss}}{P_{in}} \]

where \( P_{in} \) is input microwave power from the microwave power supply, \( P_{loss} \) is microwave power loss that was calorimetrically measured by the temperature rise of the cooling water of the reflectionless termination.

2.3 Emission Spectroscopy of the atmospheric microwave discharge plasma
The setup of the device for the spectroscopic analysis is shown in Fig. 3. The emission light from the
atmospheric microwave discharge plasma is collected by a condenser lens and introduced into a monochromator through an optical fiber. A mercury (Hg) standard light source was used for the wavelength calibration at 365.01nm. The spectroscopic analysis of the atmospheric pressure plasma generated with a mixture of Ar to 10 l/min and N₂ to 0.5 l/min was carried out.

Radiant intensity dependence measurements on N₂ concentration to Ar and spectroscopic analysis to identify constituent elements of the atmospheric microwave discharge plasma were carried out. Radiant intensity of the plasma was measured by a photo diode and is directly proportional to the amount of excited particles in the plasma.

2.4 Observation of a state of the atmospheric microwave discharge

Observation of the state of the atmospheric pressure plasma discharged by the microwave was carried out using a high-speed flaming camera at microwave input power of 573W. The working gas was a mixture of Ar at 10 l/min and N₂ at 0.5 l/min. The frame rate of the camera was 1200 fps (frames per second). The maximum frame rate of the high speed camera is 10000 fps. The luminous intensity of the atmospheric microwave discharged plasma however was insufficient to take photographs at more than 5000 fps under the experimental condition.

3. Results and Discussion

3.1 Fundamental characteristics of the atmospheric microwave discharge plasma source

The stable operating range of anode current (microwave input power) versus working gas flow rate is shown in Fig. 4. Anode currents of 160, 180, 200mA correspond to microwave powers of 455, 515, 573W respectively. At a low gas flow rate, the atmospheric pressure plasma became unstable, because the swirling flow of the working gas in the plasma torch is weakened. At a high gas flow rate, the plasma can not be maintained because of microwave power per flow rate. Electrons in the plasma accelerated insufficiently for discharging with an increase of neutral particles in the working gas.

Measured results of microwave absorption factor: ηP at several N₂ concentrations are shown in Fig. 5. The values of ηP decrease with an increase of the N₂ concentration in the working gas and are proportional to the input microwave power at each N₂ concentration.

3.2 Spectroscopic analysis results for the atmospheric microwave discharge plasma

The spectroscopic analysis results from 300 to 500nm.
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At wavelengths from 350 to 400 nm, several peaks and band spectra are found and are considered as particle spectra of N\textsubscript{2}. No atomic line of NI nor ArI was observed. The N\textsubscript{2} emission is predominantly from excited particles. The first excited state for N\textsubscript{2} is 5.23 eV. The excited N\textsubscript{2} was thought to have been ionized by a metastable Ar. Then, this atmospheric microwave discharge was thought to have been maintained by the cumulative ionization of N\textsubscript{2}. The line emission from Al, W, and Cu of which the plasma source was constructed was so weak. That implies that the plasma contains few impurities.

The measured results of radiant intensity dependence on the N\textsubscript{2} concentration to Ar are shown in Fig. 7. The figure indicates that radiant intensity of the atmospheric microwave discharge plasma increased with 500 nm are shown in Fig. 6. At wavelengths from 350 to 400 nm, several peaks and band spectra are found and are considered as particle spectra of N\textsubscript{2}. No atomic line of NI nor ArI was observed. The N\textsubscript{2} emission is predominantly from excited particles. The first excited state for N\textsubscript{2} is 5.23 eV. The excited N\textsubscript{2} was thought to have been ionized by a metastable Ar. Then, this atmospheric microwave discharge was thought to have been maintained by the cumulative ionization of N\textsubscript{2}. The line emission from Al, W, and Cu of which the plasma source was constructed was so weak. That implies that the plasma contains few impurities.

The measured results of radiant intensity dependence on the N\textsubscript{2} concentration to Ar are shown in Fig. 7. The figure indicates that radiant intensity of the atmospheric microwave discharge plasma increased with
the increase of N₂ concentration in the working gas. The radiant intensity at 40% of N₂ is three times larger than that at 20% of N₂.

3.3 Observation of the state of atmospheric microwave discharge.

Periodic blinking of the atmospheric microwave discharge plasma generated by our plasma source was found from the photographs taken by using the high speed camera. A series of states of microwave discharge are shown in Fig. 8. The frequency of plasma blinking was 120Hz because the brightest flame was seen at every ten frames at 1200 fps repeatedly. The microwave frequency of 2.45GHz is too high to cause the blinking at 120 Hz. A current waveform to the anode of the magnetron in the microwave power supply was observed and has a ripple of 120 Hz caused by full-wave rectification of the commercial power of single phase 60Hz, because the DC power supply for the anode of the magnetron is not stabilized. As the microwave power depends on the anode current directly, the blinking of the atmospheric plasma is thought to be caused by the ripple of the anode current.

4. Conclusion

The atmospheric microwave discharge plasma source was fabricated and its fundamental characteristics were investigated with a working gas mixture of N₂ and Ar gases.
(1) An atmospheric pressure microwave discharge plasma was generated with a mixed working gas of Ar and N₂ in the plasma source. This is similar to the Penning effect.
(2) The microwave absorption ratio of the atmospheric microwave discharge plasma decreased with an increase of N₂ percentage in the working gas. It was proportional to the input microwave power at each N₂ concentration.
(3) Radiant intensity of the atmospheric microwave discharge plasma increased with an increase of N₂ percentage in the working gas.
(4) By spectroscopic analysis, it was found that degree of ionization in the atmospheric microwave discharge plasma was low and the emission was mainly from N₂ particles up to the input microwave power of 600W.
(5) The generated atmospheric microwave discharge plasma contained few impurities because of the electrodeless discharge.

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