Optical Emission Characteristics of Atmospheric-Pressure Nonequilibrium Microwave Discharge and High-Frequency DC Pulse Discharge Plasma Jets

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Optical Emission Characteristics of Atmospheric-Pressure Nonequilibrium Microwave Discharge and High-Frequency DC Pulse Discharge Plasma Jets

Toshifumi Yuji, Shuitsu Fujii, Narong Mungkung, and Hiroshi Akatsuka

***Abstract*—With an aim to understand the mechanism of surface processing by atmospheric-pressure nonequilibrium discharge plasma jets, we measured the vibrational and rotational temperatures in the plasmas by means of optical emission spectroscopy (OES) measurement method. This paper focuses on the OES measurement method using a torch-shaped atmospheric-pressure nonequilibrium discharge plasma jet power supply consisting of a microwave (2.45-GHz) generator and a high-frequency (5.0–10-kHz) dc pulse power supply, using a gas mixture of Ar (8.0 L/min) and N_2 (0.1–0.5 L/min) as the discharge plasma gas, and changing the flow rate of N_2 gas at the input power of 100–150 W. Upon comparing vibrational and rotational temperatures (0.18–0.27 eV) determined from the OES measurement method using two types of atmospheric-pressure nonequilibrium discharge plasma jets, results indicate that the microwave discharge plasma jet has considerably low vibrational and rotational temperatures.**

***Index Terms*—$Ar + N_2$ mixture gas, high-frequency dc pulse, microwave, plasma jet, vibrational and rotational temperatures.**

I. INTRODUCTION

A GREAT deal of research work on surface modification and surface treatment technology for plastic films using plasma has been carried out in low-pressure vacuums [1]–[4]. Up to now, however, there were many disadvantages in treatment in vacuums, such as the complexity of the equipment and restrictions on the amount or size of the object treated. In recent years, atmospheric-pressure nonequilibrium discharge plasma technology has been proposed, and since this technology has many advantages, such as low costs, and to be set up easily by using existing plasma power supply equipment, there have been many reports on application examples for surface modification of metallic materials and as surface cleaning technology for semiconductor materials [5]–[8].

With regard to such surface modification and surface treatment technology based on plasma, the plasma gas temperature and the existence of radical species in vibrational-excited states in the plasma are important factors. The vibrational-excited level of the radical species, as well as the vibrational and rotational temperatures of the plasma gas, is an important parameter in establishing the best conditions for plasma-to-surface treatment [10], [11]. However, since atmospheric-pressure nonequilibrium discharge plasma has significantly low gas temperature in comparison with electron temperature, various plasma parameters, such as radical species, electron density, and electron temperature in the plasma, cannot be easily measured. A simple and accurate measurement method is therefore sought after [12]–[14].

In this paper, optical emission spectroscopy (OES) measurement method is carried out in order to understand the characteristics of atmospheric-pressure nonequilibrium discharge plasma jets excited by microwaves as well as high-frequency dc pulse power [15]. We aimed to establish plasma parameters for atmospheric-pressure nonequilibrium discharge plasma for application to actual dry process technology and to introduce dry process technology into new fields for the atmospheric-pressure nonequilibrium discharge plasma. The main characteristic of a high-frequency dc pulse power supply is that, by turning the pulse on and off, the radical generation period can be adjusted, and this, in turn, allows for developments in the plasma surface treatment process for a broad variety of materials. With regard to the comprehension of fundamental plasma characteristics, since probe measurement, etc., is difficult, we used a spectrometric system and conducted OES measurement, which is a relatively simple measurement method for determining plasma characteristics, and vibrational and rotational temperatures, as approximate values of the gas translational temperature, in the atmospheric-pressure nonequilibrium microwave discharge plasma jet using a high frequency pulsed power supply for generating plasma were experimentally determined [16]–[18].

II. MEASUREMENT EQUIPMENT AND METHOD

Fig. 1 shows an outline for the atmospheric-pressure nonequilibrium microwave discharge plasma generation equipment system and the atmospheric-pressure nonequilibrium high-frequency dc pulse discharge plasma generation equipment system (Haiden Laboratory, PJ-6K). Atmospheric-pressure

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T. Yuji is with the Faculty of Education and Culture, University of Miyazaki, Miyazaki 889-2192, Japan (e-mail: yuji@cc.miyazaki-u.ac.jp).

S. Fujii is with ADTEC Plasma Technology Company, Ltd., Fukuyama 721-0942, Japan.

N. Mungkung is with the Department of Electrical Technology Education, King Mongkut's University of Technology Thonburi, Bangkok 10140, Thailand.

H. Akatsuka is with the Research Laboratory for Nuclear Reactors, Tokyo Institute of Technology, Tokyo 152-8550, Japan.

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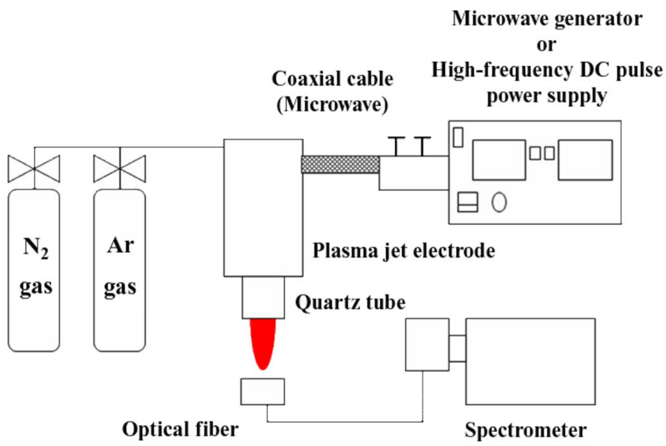


Fig. 1. Schematic of the experimental setup.

Fig. 3. Photograph of high-frequency dc pulse discharge plasma jet.

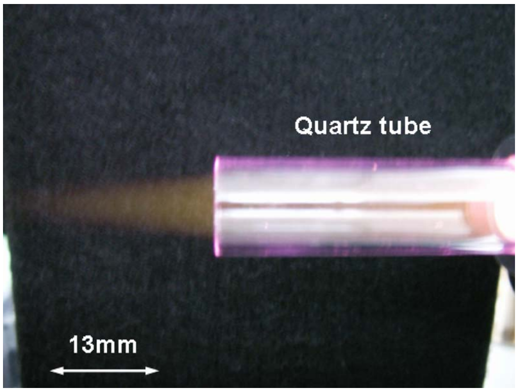


Fig. 2. Photographs of microwave discharge plasma jet.

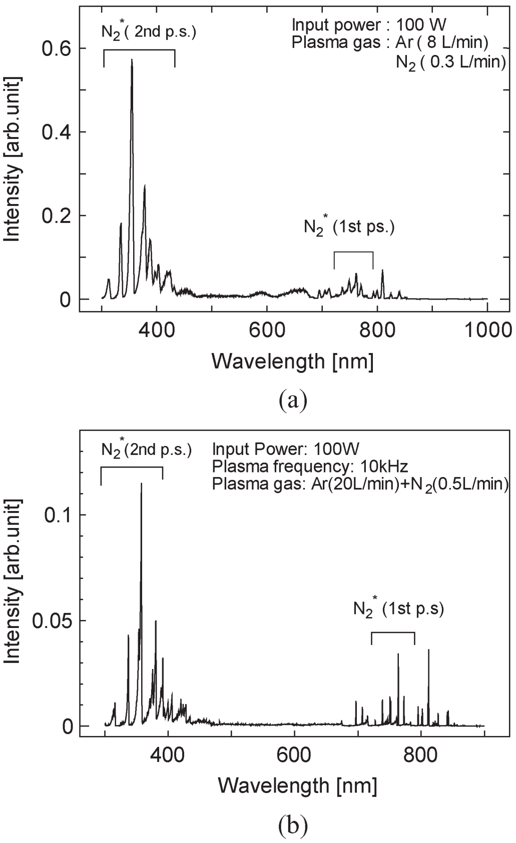
nonequilibrium microwave discharge plasma jet was generated by conducting steady discharge using an atmospheric-pressure nonequilibrium discharge plasma jet torch electrode and a microwave (2.45-GHz) generator for generating plasma [19]. For both types of equipment, a mixture of Ar gas and N_2 gas was used for the plasma gas, where the flow rate of Ar gas was kept constant at 8.0 L/min, to which the nitrogen gas was mixed in arbitrarily at a flow rate fluctuating from 0.1 to 0.5 L/min.

Fig. 2 shows a photograph of the plasma generation state of the atmospheric-pressure nonequilibrium microwave discharge plasma jet with argon (Ar) gas (8.0 L/min) and nitrogen (N_2) gas (0.5 L/min). The emission of the plasma is clear with an increase in the N_2 gas. A coaxial cable (RG-393) was used for the transmission path of the microwave. The main material for the atmospheric-pressure nonequilibrium discharge plasma jet torch is aluminum. The plasma jet torch discharge tube is quartz glass with an inner diameter of 10 mm, outer diameter of 13 mm, and length of 170 mm. The antenna for the plasma jet torch is made of aluminum and has a diameter of 7.0 mm and length of 23 mm.

Fig. 3 shows a photograph of the plasma generation state of the atmospheric-pressure nonequilibrium high-frequency dc pulse discharge plasma jet with Ar gas (8.0 L/min) and N_2 gas (0.5 L/min). In addition, we also used a high-frequency dc pulse power source for plasma generation and atmospheric-pressure nonequilibrium discharge plasma jet torch electrode to generate plasma. The plasma jet torch consisted of a titanium rod (OD: 4 mm and length: 10 mm) at the center and a SUS pipe surrounding the rod. In the upper part of the plasma jet torch, a quartz tube (OD: 26 mm, ID: 24 mm, and length: 87 mm) was placed between the titanium rod and the SUS pipe (OD: 36 mm, ID: 30 mm, and length: 87 mm), generating dielectric barrier discharges to produce reactive species [20]. The ID and length of the plasma jet torch were 10 and 20 mm, respectively. In the lower part of the torch where dielectrics are not introduced, arc discharges were generated. By using the electromagnetic pumping effect, the reactive species generated in the upper part of the plasma jet torch were effectively emitted.

In the OES measurement method, we used a monochromator (Horiba, HR320) with an optical fiber as a light guide from the plasma. The slit width of the monochromator was set at 125 μm at the entrance port and 175 μm at the exit port. The slit height was set at 100 μm at both the entrance port and the exit port. The wavelength resolution was 0.1 nm. A photomultiplier was used as a detector. The OES measurement method was conducted using a monochromator with a scanning mechanism after plasma was generated stationarily under stable conditions. The optical fiber head was placed 35 mm in the axial direction from the plasma jet torch, and a quartz plate was set between the fiber head and the plasma jet torch to avoid thermal damage. The gas translational temperature of the N_2 gas plasma can be well approximated by the rotational temperature of nitrogen molecules under atmospheric-pressure nonequilibrium discharge plasma [21]. We determine the rotational temperature from the observed spectrum of the second positive system.

Fig. 4 shows an example of the optical emission spectra when the plasma gas consisted of a mixture of Ar gas (8.0 L/min) and N_2 gas (0.3 L/min), with a wavelength of 300–900 nm. Due to sufficient intensity in the transition from the $C^3\Pi_u$ state to the $B^3\Pi_g$ state, an emission spectrum of a second positive system was obtained. In order to clarify the state of vibrational kinetics and to understand vibrational temperature, we used a spectroscopic examination to measure the relative population density of the vibrational-excited state of $N_2C^3\Pi_u$ with the vibrational quantum number $v = 0 - 4$. The gas translational



(a)

(b)

Fig. 4. Emission spectrum in 300–900 nm of atmospheric-pressure nonequilibrium Ar + N_2 mixture gas discharge plasma jet. (a) Microwave discharge plasma jet. (b) High-frequency dc pulse discharge plasma jet.

temperature of the nitrogen plasma can be closely approximated by the rotational temperature of nitrogen molecules under an atmospheric-pressure nonequilibrium discharge. Rotational temperature is obtained from a comparison of the second positive spectra observed experimentally with the calculated ones [22]. As described in [23] in detail, the vibrational and rotational temperatures can be obtained by calibrating the sensitivity of the spectra, fitting them through theoretical calculations using the vibrational and rotational temperatures as parameters, and applying them as the best fit parameters.

Fig. 5 shows an example of the comparison. The gas temperature of plasmas can be approximated by the rotational temperature. Let us discuss the effect of some broadening mechanism. The Doppler broadening can be estimated from the ratio of the thermal velocity v_{th} to the light velocity c by the following equation:

$$\frac{\Delta\lambda_D}{\lambda} = \frac{v_{\mathrm{th}}}{c} \tag{1}$$

where $\Delta\lambda_D$ is the Doppler width. Equation (1) leads to the Doppler broadening of about $10^{-6}\lambda$, which is much smaller than the instrumental resolution. We may have to consider the Doppler blue shift since we measure the plasma from the downstream direction. However, the effect of the Doppler blue shift is also imperceptible, because the flow velocity of the present plasma jet is much smaller than the sound velocity, and consequently, it is also negligible.

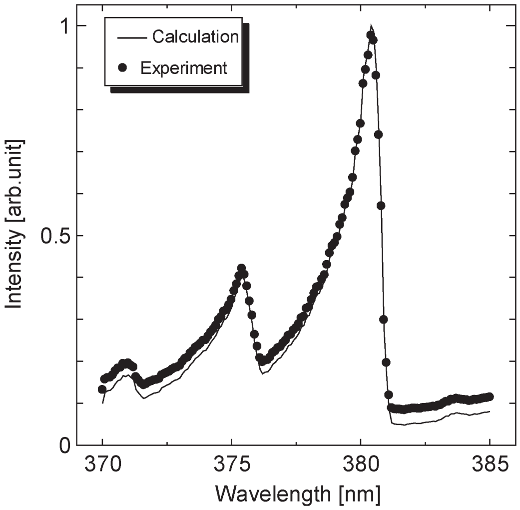


Fig. 5. Example of fitting operation of spectrum of N_2 second positive system measured experimentally by the one calculated theoretically (high-frequency dc pulse discharge plasma jet).

Meanwhile, the pressure broadening $\Delta\lambda_P$ is estimated as follows, since the neutral–neutral collision is the predominant process to the excited nitrogen molecule under the present condition:

$$\frac{\Delta\lambda_P}{\lambda} = \frac{\nu_{\mathrm{col}}}{\nu} = \frac{N\sigma v_{\mathrm{th}}}{\nu} \tag{2}$$

where N is the number density of the neutral molecules, σ is the neutral–neutral collision cross section, and ν is the frequency of the emitted light. Equation (2) shows that the pressure broadening becomes about $10^{-7}\lambda$, which is also much smaller than the present experimental resolution. In consequence, the spectral resolution of the present detection system is determined from the equipment width and estimated to be about 0.1 nm. In this way, we can fit the emission spectrum of the second positive system obtained experimentally by the theoretical calculation with sufficient accuracy.

III. RESULTS AND DISCUSSION

Fig. 6 shows the relationship between the vibrational and rotational temperatures in atmospheric-pressure nonequilibrium microwave discharge plasma jet when a mixture of Ar gas with a constant flow rate of 8.0 L/min and N_2 gas with a flow rate ranging from 0.1 to 0.5 L/min was used as the plasma gas, at plasma input powers of 100 and 150 W. From this figure, it can be confirmed that increases in the flow rate of N_2 gas and plasma input power cause an increase in the vibrational and rotational temperatures. The increase in the flow rate of N_2 gas represents an increase in the density of nitrogen molecules, which then cause a large number of vibrational-excited states to be generated. Afterward, collisional relaxation states are generated in between the vibrational-excited states, and the vibrational temperature increases. For example, Koike *et al.* [24] speculate that with low-pressure N_2 gas discharge plasma jets, in regard to stationary distribution of electron temperature, electron energy distribution function, gas temperature, gas density, and dissociation degree, the density distribution

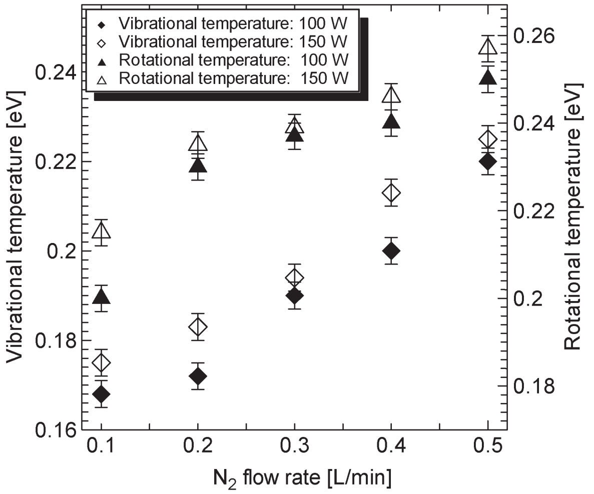


Fig. 6. Relationship between input power and vibrational and rotational temperatures at atmospheric-pressure nonequilibrium Ar and N_2 gas microwave discharge plasma jet measured by OES.

of vibrational-excited levels for nitrogen in plasma is sought after as a nonlinear simultaneous differential equation up until the achievement of a steady state under appropriate initial conditions. Based on the results of this density distribution of vibrational-excited states, high densities of vibrational-excited states indicate the high rate of reactivity, and generation of radicals and radical reactions during the plasma gas phase occur actively. In proportion to the increase in nitrogen molecules in the second positive system that accompanies an increase in the flow rate of N_2 gas, the vibrational-excited density also increases. Due to an increase in the frequency of collisions between nitrogen molecules, the VT process becomes active, and the rotational temperature is also thought to increase. With the atmospheric-pressure nonequilibrium microwave discharge plasma jet using a microwave power supply, if the flow rate of N_2 gas is changed when the input amount is 100 W, the vibrational temperature in the plasma is approximately 0.18–0.24 eV (2090 K–2800 K), whereas the rotational temperature is approximately 0.21–0.26 eV (2440 K–3020 K), and it was confirmed that the plasma is of a relatively low temperature. With an increase in the flow rate of N_2 gas, both the vibrational and rotational temperatures increase as well. The reason behind this is that vibrational–vibrational energy exchange collisions (VV process) and vibrational–translational energy relaxation collisions (VT process) [25] for nitrogen molecules occur frequently in atmospheric-pressure nonequilibrium microwave discharge plasma, and ultimately, both the vibrational and rotational temperatures increase. Fundamentally, the following is considered as the reaction process for these radicals [26]:

$$N_2^*(\text{low level}) + N_2^*(\text{low level}) \rightarrow N_2 + N_2^*(\text{higher level}) \quad (3)$$

$$N_2^*(\text{low level}) + X \rightarrow N_2^*(\text{low level}) + X(\text{or } X^*) \quad (4)$$

$$\rightarrow N_2 + X + hv \quad (5)$$

$X = N_2$, Ar, and so on.

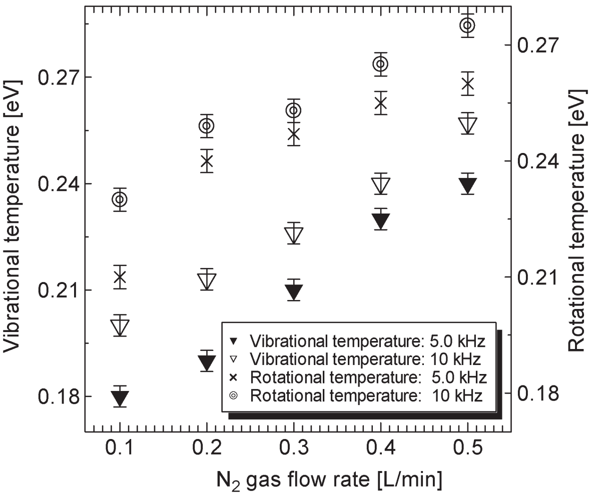


Fig. 7. Relationship between N_2 gas flow rate and vibrational and rotational temperatures at atmospheric-pressure nonequilibrium $Ar + N_2$ mixture gas high-frequency dc pulse discharge plasma jet measured by OES.

A summary of this section is given hereinafter. In order to investigate the characteristics in the plasma of an atmospheric-pressure nonequilibrium microwave discharge plasma jet using microwave generator, the vibrational and rotational temperatures in the plasma gas were experimentally calculated. From these results, it was discovered that by changing the flow rate of N_2 gas, the vibrational and rotational temperatures in the plasma gas also change. Temperatures of approximately 0.18–0.26 eV (2090 K–3020 K) were obtained for the vibrational and rotational temperatures in the plasma gas, and it was also understood that atmospheric-pressure nonequilibrium plasma jet is of a relatively low temperature.

Fig. 7 shows the relationship between vibrational and rotational temperatures in high-frequency dc pulse discharge plasma jet when a mixture of Ar gas with a constant flow rate of 20 L/min and N_2 gas with a flow rate changing from 0.1 to 0.5 L/min was used as the plasma gas, at two different high-frequency dc pulse frequencies of 5.0 and 10 kHz and a plasma input power of 100 W. From this graph, it can be confirmed that an increase in the flow rate of N_2 gas results in an increase in the vibrational and rotational temperatures. In addition, the vibrational and rotational temperatures also increase when the dc pulse frequency is changed from 5.0 to 10 kHz. For the high-frequency dc pulse power supply for plasma generation that was used in this experiment, keeping the plasma input power constant and changing the dc pulse high frequency in order to control the plasma input power through a constant voltage control system can be considered as having contributed to the increase in vibrational and rotational temperatures. This is based on the fact that the formula that is indicated as a fundamental characteristic formula (6) for high-frequency dc pulse power supply used for plasma generation is realized [27]

$$W = F \int^{t+T} I(t)V(t)dt. \quad (6)$$

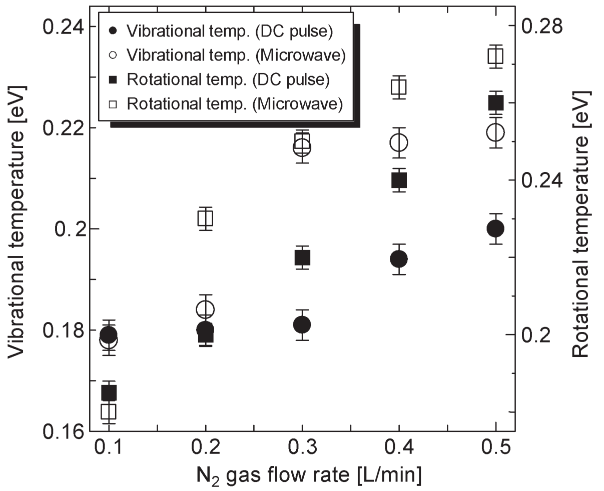


Fig. 8. Relationship between input power and vibrational and rotational temperatures at atmospheric-pressure nonequilibrium Ar and N_2 gas (microwave and high-frequency dc pulse) discharge plasma jets measured by OES.

In this formula, W is the plasma input power, I is the dc pulse current, V is the high-frequency dc pulse voltage, F is the plasma dc pulse frequency, t represents the time change for high-frequency dc pulse voltage, and T is the pulse period. From the same figure, it can be confirmed that an increase in the flow rate of N_2 gas and an increase in the plasma input power cause increases in the vibrational and rotational temperatures. In addition, at a dc pulse frequency of 5.0 kHz, the vibrational temperature was between 0.18 and 0.24 eV (2090 K–2790 K), and the rotational temperature was between 0.21 and 0.26 eV (2440 K–3020 K). At a plasma pulse frequency of 10 kHz, the vibrational temperature ranged from 0.20 to 0.26 eV (2320 K–3020 K), and the rotational temperature ranged from 0.23 to 0.28 eV (2670 K–3200 K). This confirms that by increasing the dc pulse high frequency, the vibrational and rotational temperatures in the plasma also increase. The main reason behind this phenomenon is that, in accordance with an increase in the flow rate of N_2 gas, the densities of various nitrogen molecules in vibrational states and, in particular, vibrational-excited states increase. This, in turn, causes an increase in the collision frequency of vibrational-excited nitrogen molecules, and due to an increase in the VV exchange process and VT relaxation process, increases in the vibrational and rotational temperatures can be conjectured. In addition, for nitrogen plasma at atmospheric pressure, it is necessary to give consideration to the production and loss process of excited particles in an excited state. When investigating only nitrogen molecules with respect to the aforementioned topics, the electron impact excitation/deexcitation (Ve process), vibrational–vibrational energy migration process (VV transfer), and vibrational–translational energy relaxation process (VT transfer) are mainly thought to be important [14].

Fig. 8 shows a comparison between the two types of equipment—atmospheric-pressure nonequilibrium high-frequency dc pulse discharge plasma jet and atmospheric-pressure nonequilibrium microwave discharge plasma jet—with regard to vibrational and rotational temperatures in plasma jets obtained by the OES measurements, when a mixture of Ar gas with a flow rate of 8.0 L/min and N_2 gas with a flow rate ranging from 0.1 to 0.5 L/min was used as the plasma gas at a plasma input power of 100 W. For the high-frequency dc pulse discharge plasma jet, the dc pulse frequency was 5.0 kHz. The maximum allowable pulse frequency for the dc pulse discharge plasma jet is 10 kHz. As it is impossible to maintain a plasma input power of 100 W when the pulse frequency becomes greater than 5.0 kHz, the plasma input power becomes at least 150 W when the plasma frequency is greater than 5.0 kHz. On the other hand, with the atmospheric-pressure nonequilibrium microwave discharge plasma jet, if a plasma input power that exceeds 150 W is used, the torch electrode becomes broken due to the structure of the electrode. As a result, it was decided to conduct a research using a plasma input power of 100–150 W in this experiment.

From the same graph, it can be seen that, in the atmospheric-pressure nonequilibrium high-frequency dc pulse discharge plasma jet, the vibrational temperature in the plasma was 0.19–0.26 eV, and the rotational temperature was 0.18–0.27 eV at a pulse frequency of 5.0 kHz. In the atmospheric-pressure nonequilibrium microwave discharge plasma jet, however, the vibrational temperature was 0.18–0.20 eV, and the rotational temperature was 0.18–0.22 eV. When comparing the vibrational and rotational temperatures in atmospheric-pressure nonequilibrium high-frequency dc pulse discharge plasma jets and in atmospheric-pressure nonequilibrium microwave discharge plasma jets, there were a maximum difference of approximately 0.02 eV for vibrational temperature and a maximum difference of approximately 0.03 eV for rotational temperature. It was confirmed that vibrational and rotational temperatures in the plasma in atmospheric-pressure nonequilibrium microwave discharge plasma jets were relatively lower.

IV. CONCLUSION

Upon comparing the atmospheric-pressure nonequilibrium high-frequency dc pulse discharge plasma jet with the atmospheric-pressure nonequilibrium microwave discharge plasma jet in terms of characteristics, it was found that since the atmospheric-pressure nonequilibrium high-frequency dc pulse discharge plasma jet is also structurally equipped with a mechanism for generating arc discharge, the radical density in the plasma jet is low due to the impact of heat, and as there is, or will continue to be, a migration to thermal plasma, the plasma is thought to be in a state close to equilibrium.

The reason for these phenomena can be considered as follows: The increase in the flow rate of nitrogen gas leads to an increase in the nitrogen molecule density in the discharge region. This increases the collision frequency between electrons and neutrals, and as a result, more vibrationally excited states are generated. Then, collisional relaxation becomes more frequent between vibrationally excited states, i.e., the VV energy transfer, which finally leads to an increase in vibrational temperature.

For the atmospheric-pressure nonequilibrium microwave discharge plasma jet, the vibrational and rotational temperatures are lower than with the atmospheric-pressure nonequilibrium high-frequency dc pulse discharge plasma jet, and it is speculated that high-density radicals [28] are being generated in the plasma jet.

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Toshifumi Yuji was born in Miyazaki, Japan, on June 2, 1976. He received the M.E. degree from the University of Miyazaki, Miyazaki, in 2003, and the Ph.D. degree from Tokyo Institute of Technology, Tokyo, Japan, in 2007.

He became a Research Associate with Hiroshima National College of Maritime Technology in 2005 and an Assistant Professor with Oita National College of Technology in 2006. He is currently a Lecturer with the Faculty of Education and Culture, University of Miyazaki. His research interests include plasma application and electric power installation.

Dr. Yuji is a member of the Institute of Electrical Engineers of Japan and the Japan Society of Applied Physics.

Shuitsu Fujii was born in Hiroshima, Japan, on August 16, 1948. He received the B.S. degree in telecommunication engineering from Osaka Electro-Communication, Osaka, Japan, in 1972.

Since 1997, he has been the CEO of ADTEC Plasma Technology Company, Ltd., Fukuyama, Japan. His research interests include microwaves as a whole.

Narong Mungkung was born in Lopburi, Thailand, in 1965. He received the B.Sc. Ind. Ed. degree in electrical engineering from the Department of Electrical Technology Education, King Mongkut's University of Technology Thonburi, Bangkok, Thailand, the M.Tech. Ed. degree in electrical technology from King Mongkut's Institute of Technology North Bangkok, Bangkok, and the M.Eng. and D.Eng. degrees in engineering from Nippon Institute of Technology, Saitama, Japan, in 2000 and 2003, respectively.

He was JICA participant of HITC and Polytechnic University, Japan. He is currently with the Department of Electrical Technology Education, King Mongkut's University of Technology Thonburi. His research interests include plasma application, energy conservation, and engineering education.

Hiroshi Akatsuka was born in Kyoto, Japan, on December 27, 1962. He received the M.E. degree from Kyoto University, Kyoto, in 1987, and the Ph.D. degree from Tokyo Institute of Technology, Tokyo, Japan, in 1995.

He was with NEC Corporation from 1987 to 1991 and moved to the Research Laboratory for Nuclear Reactors, Tokyo Institute of Technology, as a Research Associate, in 1991. He has been an Associate Professor with the Research Laboratory for Nuclear Reactors and the Department of Energy Sciences, Tokyo Institute of Technology, since 1995. His research interests include atomic and molecular processes in plasmas, plasma diagnostics, and spectroscopy.

Dr. Akatsuka is a member of the Institute of Electrical Engineers of Japan, the Japan Society of Applied Physics, the Physical Society of Japan, the Japan Society of Plasma Science and Nuclear Fusion Research, the Spectroscopical Society of Japan, the Laser Society of Japan, Atomic Energy Society of Japan, Combustion Society of Japan, and the Visualization Society of Japan.