Investigation of Water-Vapor Plasma Excited by Microwaves as Ultraviolet Light Source

Jun-Seok Oh, *Member, IEEE*, Kazumasa Kawamura, Bimal K. Pramanik, and Akimitsu Hatta

***Abstract*—The potential of using water-vapor plasmas excited by microwaves as a ultraviolet (UV) light source has been investigated by using various pressures and input powers. The UV irradiation power increased and saturated at a pressure range dependent on the input power. On the other hand, other visible and infrared emissions corresponding to four atomic lines, i.e., the Balmer series of hydrogen at 486.1 nm (H_β) and 656.3 nm (H_α) and oxygen atoms at 777.2 and 844.6 nm, were clearly decreased with an increase in the total gas pressure. It was found that pressures (1.4–2.0 kPa) near the saturated water-vapor pressure were found to give the most intense UV irradiation. With a vapor pressure of 1.6 kPa and a total microwave power of 300 W, the power density of UV (Γ_{uv}) was measured to be 10.5 $\mu W \cdot cm^{-2}$ at a distance of 30 cm from the center of the discharge tube as measured through an optical viewing port on the cavity discharge applicator. This value for (Γ_{uv}) is comparable to that for a mercury lamp. However, the (η_{uv}) efficiency was estimated to be considerably lower than that of a mercury lamp.**

***Index Terms*—Absolute spectrum calibration, microwave, OH radical, optical-emission spectroscopy (OES), rotational temperature, saturated water-vapor pressure, ultraviolet (UV), water-vapor discharge.**

I. INTRODUCTION

ULTRAVIOLET (UV) radiation from OH radicals produced by water-vapor (H_2O) discharge was first reported in the 1920s [1]. There are two UV band systems: the 306.4-nm system ($A^2\Sigma^+ - X^2\Pi$) and the Schüler–Michel–Benoist system ($C^2\Sigma^+ - A^2\Sigma^+$) [2]. The 306.4-nm system is a well-known band that is easily observed. It extends from 244.4 to 402.2 nm and has several intense peaks concentrated in the range 302.1–308.9 nm. The other UV system is very weak and is in the high-frequency region of water-vapor discharge. Six UV emission peaks have been reported in the wavelength range 224.9–268.3 nm [2]. Out of the many UV emission peaks of water-vapor discharge, those in the UV-B region (i.e., mid-UV: ∼280–320 nm) are of particular interest [3], [4], since these wavelengths are anticipated to be useful for producing high-durability products such as automotive exterior coatings and roofing materials and also for sterilizing medical equipment [5], [6]. Furthermore, water vapor is considerably cheaper and easier to handle than high-quality gas mixtures of hydrogen and oxygen.

However, some difficulties need to be overcome before UV emission from water-vapor discharge plasmas can be applied. One of these problems is the strong chemical reaction that occurs between these plasma and the surfaces of the metal electrodes of conventional direct- and alternating-current power sources. To avoid major problems when using conventional operating methods, the plasma should make no contact with the electrodes. This can be achieved by using electrodeless discharge plasmas, such as radio-frequency (RF) plasmas either ICPs or CCPs dielectric-barrier-discharge (DBD) plasmas generated using an external electrode, and plasmas produced by 2.45-GHz microwaves generated by widely used magnetrons or recently developed solid-state microwave power sources. For an example, electrodeless microwave water-vapor discharges have also been created at atmospheric pressure using 2.45 GHz for space propulsion [7].

Out of these plasma sources, the conventional magnetron microwave was used to generate water-vapor plasmas because it can generate higher electron temperatures and higher plasma densities than other effect of directly heating the water molecules. It is well known that 2.45-GHz microwaves interact strongly with water molecules, so that water can be easily heated by commercial microwave ovens. These reasons led us to investigate the use of a conventional magnetron microwave to generate water-vapor plasmas.

In the first part of this paper, we investigate the emission characteristics of water-vapor discharge plasmas and calibrate the absolute spectrum using a Xe arc lamp. The accuracy of our estimation method was confirmed by conducting a comparative study using a low-pressure mercury germicidal lamp as a reference. We then estimated the UV power density (Γ_{uv}), total UV power (P_{uv}), and UV efficiency (η_{uv}) for various microwave powers and water-vapor pressures in the microwave cavity relative to those obtained using the mercury lamp. In the second part of this paper, we evaluate the direct heating effect by calculating the rotational temperature (T_R) of OH radicals [8]. Finally, we discuss the possibility of using water-vapor plasmas as UV-B light sources.

II. EXPERIMENTAL PROCEDURES

We used a conventional magnetron microwave power source that is capable of providing a maximum power of 1.5 kW and a center frequency of 2.45 GHz. It uses a WRJ-2 waveguide circuit that consists of an isolator, a directional coupler, a reducer, and an autotuner. A 450-mm-long cylindrical quartz

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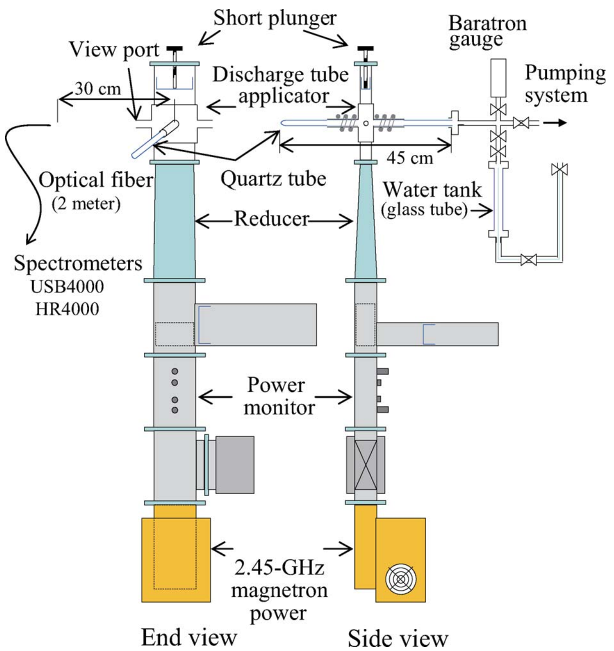

Fig. 1. Schematic of the experimental setup for water-vapor plasma excited by microwaves.

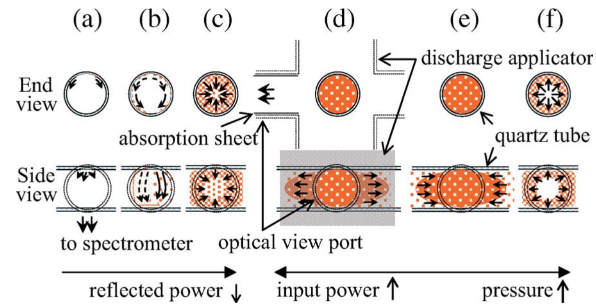

Fig. 2. Microwave discharge-plasma modes classified based on their appearance. (a) Ignition mode. (b) Along-the-wall diffusion mode. (c) Toward center expansion mode. (d) Volume-expansion mode. (e) Volume-reduction mode. (f) Toward-the-wall reduction mode. The mode was strongly dependent on the gas pressure and input power.

(SiO_2) tube having an inside diameter of 13 mm and an outside diameter of 15 mm was used for microwave discharge of water vapor. As shown in Fig. 1, this tube was attached to a cavity discharge applicator terminated with a short plunger and was connected to a microwave cavity consisting of a flat rectangular waveguide $(27 \times 96\ \mathrm{mm}^2)$. One side of the discharge tube was sealed with a quartz window that permitted the interior of the discharge tube to be observed, and the other side was connected to a vacuum system. It was evacuated using a conventional rotary pump that produced an initial vacuum of a few Pascals.

The quartz discharge tube and the water container were evacuated, and the water tank was filled with distilled water. Water vapor was spontaneously evaporated into the discharge tube until the saturated water-vapor pressure ($\sim$2.2 kPa) at room temperature (RT: $\sim$292 K) was attainted. This was achieved without using any water heating or cooling systems. This process of filling with water vapor and evacuating was repeated to eliminate any residual air from the water container. The air was deemed to have been removed from the water container when the emission peaks of the second positive system of nitrogen (280–498 nm) were not visible [2]. In the main experiment, the starting pressure was adjusted by repeatedly pumping and refilling. The introduction valve was closed once the initial pressure (P_i) had been reached. This procedure enabled us to obtain a clear discharge spectrum of water vapor without any nitrogen peaks.

The emission spectrum from the discharge was monitored using two multichannel fiber-optic spectrometers (OceanOptics, USB4000 for 200–880 nm and HR4000 for 200–420 nm) with a 2-m quartz fiber. The fiber was attached at 30-cm intervals to the central position on a cylindrical optical viewing port on the sidewall of the waveguide discharge applicator. All the emission spectra were measured under the same conditions and for the same configuration so that their relative emission intensities could be compared.

III. Results and Discussion

A. Formation of Water-Vapor Discharge Plasma

The formation of the water-vapor discharge plasma by microwave excitation was carefully observed under the optimal measurement conditions. After the plasma was ignited, it was localized near the inner surface of the quartz tube with a large reflected power [see Fig. 2(a)]. As the reflected microwave power decreased (or as the input power was increased), it rapidly diffused along the inner surface of the quartz tube as shown in Fig. 2(b). Finally, we were able to achieve a stable discharge as shown in Fig. 2(c)–(f). These plasmas appeared very stable at a low reflected microwave power. Following all the spectra were obtained under these stable-discharge conditions. The plasma formation can be controlled by two major parameters: gas pressure and input power. We assigned names to the possible plasma modes (see caption of Fig. 2).

B. Typical Emission Spectra of Water-Vapor Plasma

In our previous reports [9], [10], only four sharp emission peaks were observed in the range from 200 to 880 nm for a total gas pressure of a few hundred Pascals and a power of $\sim$150 W. These are the peaks at 308.9 nm from OH radicals, 656.3 nm (H_α) and 486.1 nm (H_β) from atomic hydrogen and 777.2 nm from atomic oxygen. We also found that the emission intensity depended on the gas pressure. Specifically, when the total gas pressure was increased, the emission intensity of the atomic lines decreased dramatically. A similar trend for the atomic oxygen line was recently reported by Hayashi *et al.* [6] for an oxygen and water-vapor RF plasma. By contrast, the band-emission intensity of OH radicals increases as the pressure is increased up to about 400 Pa, at which it became saturated and it decreased slightly at high pressures. This saturation and subsequent reduction in the intensity might be caused by a reduced input power density, such as is the case shown in Fig. 2(f). It could be possible to enhance the band-emission intensity by suppressing the peaks of the other atomic lines.

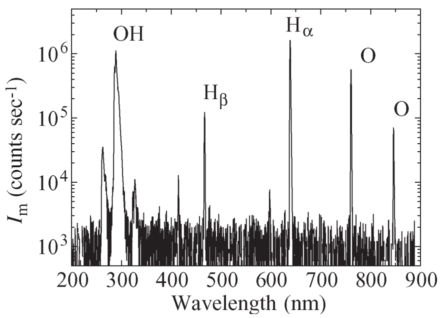

Fig. 3. Typical emission spectrum for a 2.45-GHz microwave-excited water-vapor discharge measured at a working pressure of 1.6 kPa (12 torr) with a microwave power of 300 W on a logarithmic scale.

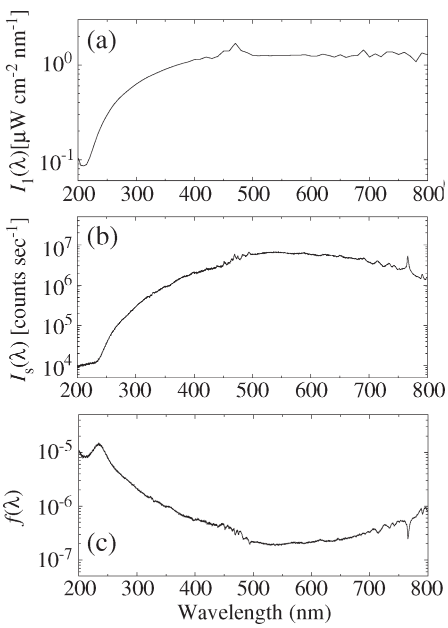

Fig. 4. (a) Spectral irradiance I_l of a standard Xe arc lamp given in the datasheet provided by the manufacturer (Hamamatsu Photonics L7810-02). (b) Emission spectrum I_s of the Xe lamp measured by spectrometer at a distance of 50 cm. (c) Correction factor $f(\lambda)$ for the absolute spectrum.

In order to achieve a strong UV output, we investigated the pressure dependence for various input powers. A typical emission from the water-vapor discharge is shown in Fig. 3 at an initial gas pressure P_i of 1.6 kPa and a power of 300 W. The pressure increased to approximately 1.7 kPa (working pressure: P_w) during the discharge. The intensities of the emission peaks were strongly enhanced, in a manner similar to that found in our previous results [9], [10]. In particular, the atomic oxygen line at 777.2 nm was remarkably enhanced, and another atomic oxygen line at 844.6 nm was clearly observed in this paper.

C. Absolute Spectrum Calibration

We calibrated the absolute spectral sensitivity of the spectrometer to obtain the absolute spectrum. This was preparatory for obtaining P_{OH} and η_{OH} of UV radiation emitted by water-vapor discharge. The absolute spectrum and the wavelength were calibrated using a standard Xe arc lamp (Hamamatsu Photonics, L7810-02) and a conventional low-pressure mercury lamp, respectively. Fig. 4(a) shows the power spectrum for the

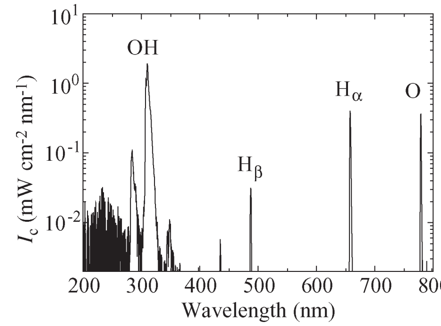

Fig. 5. Absolute spectrum of the emission spectrum I_c shown in Fig. 3 obtained by using the correction factor $f(\lambda)$.

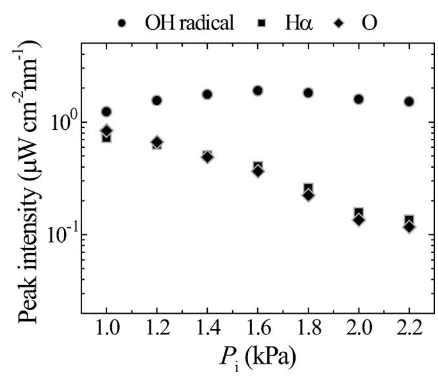

Fig. 6. Dependence of the peak intensities as a function of total gas pressure with a microwave power of 300 W (OH radicals: 308.9 nm, H_α: 656.3 nm, and O: 777.2 nm).

absolute irradiation (I_l) for the standard Xe lamp provided by the manufacturer, and Fig. 4(b) shows the spectrum I_s for the same light source measured using a spectrometer (USB4000) through a 2-m quartz fiber. We then obtained the following function $f(\lambda)$ that gives the corrected value for the absolute spectrum:

$$f(\lambda) = \frac{I_l[\text{in } \mu\text{W} \cdot \text{cm}^{-2}\text{nm}^{-1}]}{I_s[\text{in counts s}^{-1}]} \tag{1}$$

where I_s is the normalized value for 1 s. Fig. 4(c) shows a plot of $f(\lambda)$. The inverse of $f(\lambda)$ represents the optical sensitivity of the spectrometer.

Hence, by using the following expression that involves both the measured spectrum $I_m(\lambda)$ and $f(\lambda)$, we can obtain the absolute value of our measurements

$$I_c(\lambda) = f(\lambda)I_m(\lambda). \tag{2}$$

We apply this expression to the results shown in Fig. 3 to obtain I_c, which is shown in Fig. 5. In these absolute spectra results, the peak intensities of the OH radicals are an order of magnitude stronger than those of other atomic emission peaks for the present discharge conditions. This spectrum reveals the potential of water-vapor plasmas as UV source.

The other results obtained are shown in Fig. 6. The peak intensity of I_c from the OH radical increases with an increase in P_i until 1.6 kPa. However, it is nearly saturated in the vicinity of the saturated water-vapor pressure (1.4–2.0 kPa) at RT for an input power of 300 W. At even greater pressures, the intensity of the OH peak decreases slightly. This may be due to a similar reason as that for our results obtained by us previously at low input powers [9], [10]. All the atomic lines decreased with increasing pressure over the entire pressure range of 1.0–2.2 kPa.

D. UV Power Density and Total UV Irradiation Power

In this section, we estimate the UV power density OH by integrating the calibrated spectrum I_c with respect to the wavelength

$$\Gamma_{\mathrm{uv}} = \int_{\lambda_1}^{\lambda_2} I(\lambda) f(\lambda) d\lambda = \int_{\lambda_1}^{\lambda_2} I_c(\lambda) d\lambda \tag{3}$$

where $\lambda_1 = 280$ nm and $\lambda_2 = 320$ nm for the UV-B region. We also approximately estimate the total UV irradiation power (P_{OH}) from our water-vapor discharge plasma excited by microwaves. This can be calculated using

$$P_{\mathrm{OH}} = \int_s \Gamma_{\mathrm{OH}} ds. \tag{4}$$

For an accurate estimation of P_{OH}, it is necessary to measure the Γ_{OH} profile in all directions. In the present experiment, however, the emission measurement could be made only through the optical viewing port on the cavity discharge applicator. For simplicity, we assume that the plasma is a point light source and then integrate over a sphere with a distance, even though the actual plasma cylindrical. We also assumed that the power density OH was uniform over a sphere with a radius of 30 cm.

To confirm the validity of this measurement method without the cavity, we performed the same measurement for a reference mercury lamp (Mitsubishi/Osram, GL10) at a distance of 100 cm from the center of the lamp (this is the distance specified on the data sheet provided by the manufacturer). Γ_{Hg} was measured to be 21 $\mu\mathrm{W} \cdot \mathrm{cm}^{-2}$ and P_{Hg} was calculated to be 1.98 W. If we consider the number of hours that the lamp had been used, these values to not differ greatly from those specified by the manufacturers (29 $\mu\mathrm{W} \cdot \mathrm{cm}^{-2}$ and 2.7 W, respectively) [11]. This confirms that our estimation of the UV power density from the water-vapor discharge is reliable.

We now consider the effect of the microwave cavity. The maximum value of Γ_{OH} is found to be 10.5 $\mu\mathrm{W} \cdot \mathrm{cm}^{-2}$ and the corresponding value of P_{OH} is found to be 0.12 W at 30 cm from the central position of the discharge at a total pressure of 1.6 kPa and with an input power of 300 W. These values are slightly larger than those for a commercial low-pressure mercury lamp. Γ_{Hg} and P_{Hg} are measured to be 9.1 $\mu\mathrm{W} \cdot \mathrm{cm}^{-2}$ and 0.10 W, respectively, for the same configuration. The estimated values for the UV density and output power are shown in Fig. 7(a) and (b), respectively, for all cases. It is possible that the values are underestimates because the measurement was restricted to view of the plasma from the optical viewing port [see Fig. 2(d) and (e)].

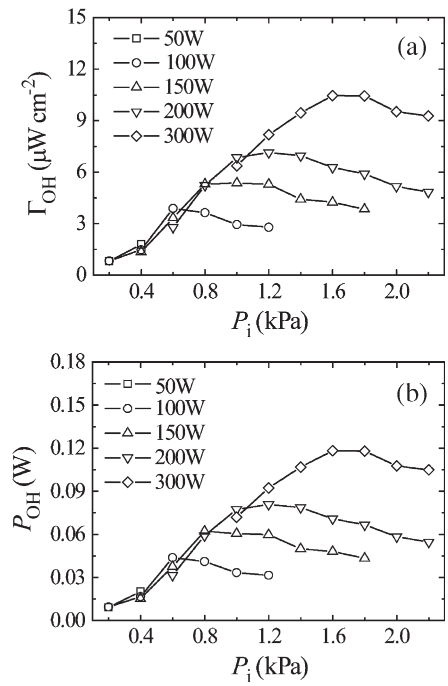

Fig. 7. Estimated (a) Γ_{OH} density and (b) P_{OH} irradiation power for UV from water-vapor discharge as a function of initial pressure P_i for various input powers.

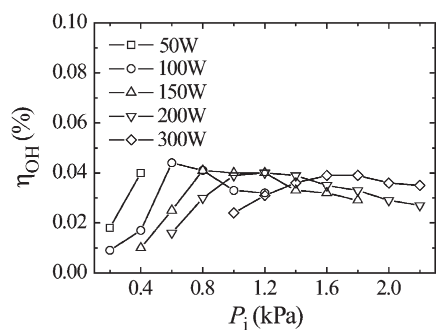

Fig. 8. Estimated UV conversion efficiency η_{OH} as a function of initial pressure P_i for various input powers.

E. Conversion Efficiency η_{uv}

The conversion efficiency of water-vapor η_{OH} is considered next. It is defined as

$$\eta_{\mathrm{uv}} = \frac{P_{\mathrm{uv}}}{P_{\mathrm{mw}}} \tag{5}$$

where P_{mw} is the input power. The efficiencies obtained are very low, being ∼0.04% in Fig. 8. This corresponds to 80% of the total output power over the measurement range of 200–800 nm. In the case of GL10, the conversion efficiency was estimated to be ∼1.03% under the same conditions in the discharge cavity. In reality, however, the mercury lamp generated a uniform plasma over the full length of the lamp. Therefore, we estimate that the conversion efficiency of the water-vapor plasma is three orders lower than that of the mercury lamp. One possible reason for this reduced efficiency could be the direct absorption of microwaves by the water molecules. This is discussed more in the following section in conjunction with a calculation of the rotational temperature (T_R).

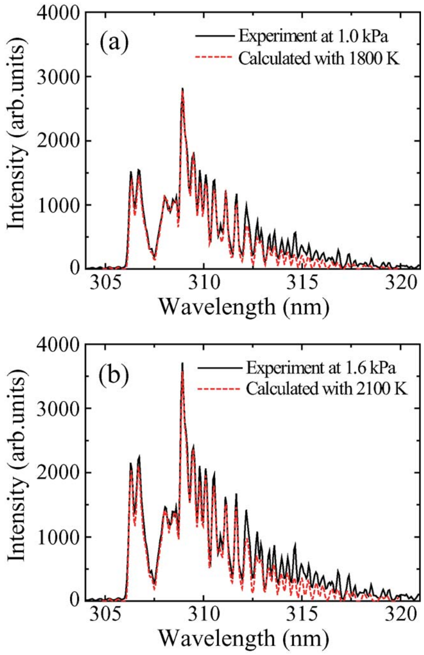

Fig. 9. Measured emission spectra from (solid line) OH radicals and (dotted line) calculated results are plotted for gas pressures of (a) 1.0 kPa (7.5 torr) and (b) 1.6 kPa (12 torr).

F. T_R of OH Radicals

The T_R of OH radicals was estimated using a calculation code [8]. This code simulates the emission spectrum for OH radicals based on T_R and the spectrometer resolution $\Delta\lambda$ (0.06 nm for HR4000). We simulated spectra by varying T_R in the range of 300–20 000 K. We then determined T_R to enable the measured spectrum to be compared with the calculated profile. Fig. 9(a) and (b) shows typical spectra for the emission peaks from the OH radicals in our measurements together with the corresponding calculation results. Based on this comparison, T_R was estimated to be 1800 K for a pressure of 1.0 kPa in Fig. 9(a) and to be 2100 K for a pressure of 1.6 kPa in Fig. 9(b). There is very good agreement between the measurement results and the calculation results.

The corresponding results are shown in Fig. 10 as a function of the water-vapor pressure P, where P_i is the initial pressure and P_w is the working pressure during discharge. T_R increases linearly with an increase in the pressure. It is also relatively high as compared to conventional low-pressure discharge and other molecular gases such as nitrogen [12]. As we anticipated, the high value of T_R is due to direct heating of the water vapor by the microwaves. These direct heating results not only in stable water-vapor plasma with a small reflected microwave power but also gives a lower UV conversion efficiency. Direct heating occurs more readily at higher pressures (or higher gas density).

IV. Concluding Remarks

Water-vapor plasmas excited by microwaves have been investigated using optical-emission spectroscopy. A clear emission spectrum was obtained and that contained four sharp atomic lines from hydrogen and oxygen, and a molecular band

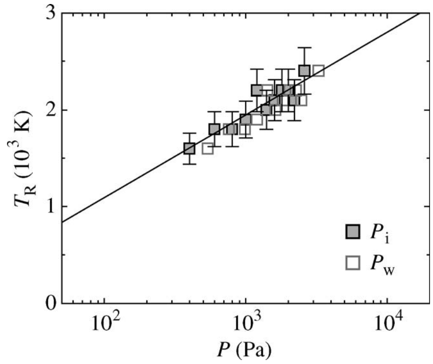

Fig. 10. Estimated values of rotational temperature T_R of OH radicals summarized by the comparison with Fig. 9. It was fitted using several values for the initial pressure (P_i) with a gradient that was calculated to be 1.6 kPa^{-1} (213 ktorr^{-1}).

emission from OH radicals in the range of 200–800 nm. The OH radical band-emission spectrum has a few tens of emission peaks in the range of 306–320 nm in the UV-B region. The intensities of the atomic lines and the molecular band are strongly dependent on the gas pressure. The peak intensities of all the atomic lines decreased drastically with increasing gas pressure. On the other hand, the value of the OH band emission increased and saturated near the saturated water-vapor pressure (1.4–2.0 kPa) at RT. In our measurements, the maximum value of OH was found to be about 10.5 $\mu\mathrm{W}\cdot\mathrm{cm}^{-2}$ at a distance of 30 cm from the central position of the discharge and at a total pressure of 1.6 kPa for an input power of 300 W. This is slightly higher than that for a mercury lamp under the same conditions. However, the UV conversion efficiency of the water-vapor plasma was estimated to be considerably lower than that of a conventional mercury lamp. This is considered to be due to the direct absorption of 2.45-GHz microwave radiation by the water molecules. In support of this, the rotational temperature (T_R) of the OH radicals was found to be high at around 1900–2100 K at the saturated water-vapor pressure. We anticipate that the UV conversion efficiency can be improved by using other methods for generating plasmas that employ electrodeless power sources, such as RF and DBD plasmas.

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