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ORIGINAL ARTICLE

Production rates and destruction frequencies of ammonia in inductively coupled $\text{H}_2\text{O}/\text{N}_2$ and H_2/N_2 plasmas

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Abstract

We compared the production rates and the destruction frequencies of ammonia in inductively coupled $\text{H}_2\text{O}/\text{N}_2$ and H_2/N_2 plasmas. We observed that the production rates of ammonia ranged between 4 and 7 $\mu\text{mol/s}$ both in the $\text{H}_2\text{O}/\text{N}_2$ and H_2/N_2 plasmas generated at rf powers between 100 and 160 W, corresponding to the energy cost of 23–25 mol/MJ. Almost the same production rates suggest the usefulness of H_2O as the resource for hydrogenation of atomic nitrogen adsorbed on surfaces. On the other hand, the destruction frequency was 2–4 times higher in the $\text{H}_2\text{O}/\text{N}_2$ than H_2/N_2 plasmas. Although the $\text{H}_2\text{O}/\text{N}_2$ plasma has a drawback in the destruction frequency, the present work shows its potential for the direct synthesis of ammonia from water with skipping the production of hydrogen.

KEYWORDS

ammonia synthesis, water vapor, hydrogen, inductively coupled plasma

1 | INTRODUCTION

Ammonia becomes more important not only as an ingredient of fertilizers but also as an energy carrier. This is because ammonia can work as a fuel for combustion [1, 2]. The combustion of ammonia does not emit carbon dioxide, which is a remarkable advantage to decelerate the global warming. However, ammonia is not perfect as a “green” energy carrier, if hydrogen, which is a raw material of ammonia, is produced from natural gas and coal, since a large amount of carbon dioxide is emitted in its production process. Ammonia can be green if hydrogen produced from water is used as the raw material, but the high production cost of “green hydrogen” prevents the industry from using it in the production process of ammonia.

The issue mentioned above is solved if we can synthesize ammonia from water. However, because of the difference in the chemical potentials of hydrogen and water ($\text{H}_2\text{O} \rightarrow \text{H}_2 + \frac{1}{2}\text{O}_2$ needs 238 kJ/mol), the synthesis of ammonia from water is difficult if we apply processes in thermodynamic equilibrium, even though catalytic and electrochemical synthesis methods have been developed to date [3–7]. The breakthrough would be possible by synthesis processes that utilize nonequilibrium plasmas. This is because the cross sections of $\text{H}_2\text{O} + e \rightarrow \text{H} + \text{OH} + e$ and $\text{H}_2 + e \rightarrow \text{H} + \text{H} + e$ are almost the same at an electron energy of 9 eV [8, 9]. In addition, the threshold electron energy of the former reaction is lower than that of the latter one. Therefore, water is promising as a hydrogen source if we apply electron impact dissociation.

In spite of the hopeful nature as a hydrogen source, the number of publications that investigate the synthesis of ammonia from nitrogen and water vapor using nonequilibrium plasmas is surprisingly small [10–15]. In addition, the synthesis rates realized to date are insufficient to consider the industrial use in future. Recently, many researchers have reported the synthesis of ammonia using liquid water and atmospheric-pressure nitrogen discharge [16–25]. However, the synthesis rates are lower than 0.1 $\mu\text{mol/s}$. Furthermore, the synthesis mechanism of ammonia in the liquid water-nitrogen discharge system has not been understood yet. For example, Gorbanev and coworkers point out the importance of the gas-phase synthesis [11], whereas Hawtof and coworkers argue the reduction of nitrogen in the liquid phase [19].

In this work, we examined the synthesis rate of ammonia in low-pressure inductively coupled plasma with the mixture of water vapor and nitrogen. We realized a synthesis rate of 4–7 $\mu\text{mol/s}$ using the low-pressure plasma generated at an rf power

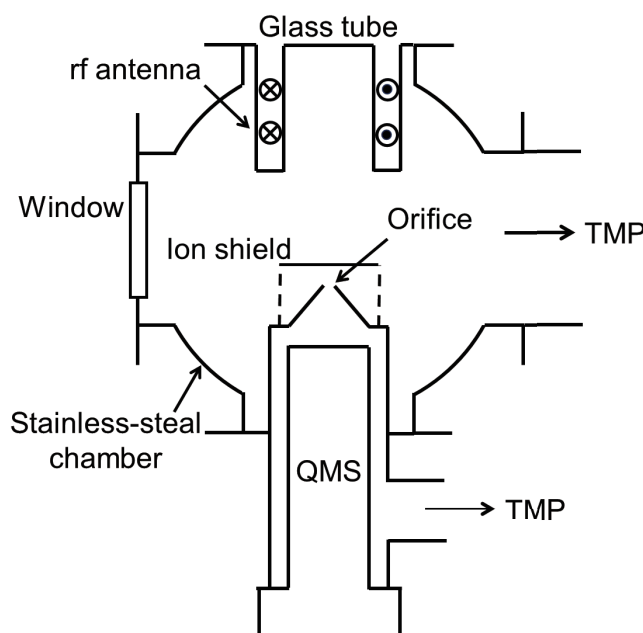


FIGURE 1 Experimental apparatus.

of 100-160 W. More importantly, this production rate was almost the same as that obtained by the plasma with the mixture of hydrogen and nitrogen. The present work shows the potential of the low-pressure $\text{H}_2\text{O}/\text{N}_2$ plasma for the economical synthesis of ammonia without emitting carbon dioxide.

2 | EXPERIMENT

The experimental apparatus is schematically shown in Fig. 1. We introduced water vapor into a stainless-steel chamber after evacuating it below 2×10^{-6} Torr using a turbomolecular pump with a pumping speed of 2000 L/s. The evacuation speed for water vapor was enhanced by installing traps at the temperature of liquid nitrogen in the vacuum chamber [26]. Liquid water was stored in a vessel connected to the vacuum chamber, and water vapor was obtained by the natural evaporation of liquid water. The flow rate of water vapor, which was controlled using a needle valve, was estimated to be 106 sccm [26]. The partial pressure of water vapor, which was measured using a capacitance manometer, was 2 mTorr. Molecular nitrogen was added to water vapor to realize a total pressure of 12 mTorr. A mass flow controller was used for feeding molecular nitrogen, and the flow rate of nitrogen was 560 sccm to obtain the partial pressure of 10 mTorr. In the experiment for the comparison, we replaced water vapor with molecular hydrogen. The partial pressures of hydrogen and nitrogen were 2 and 10 mTorr, which were obtained at flow rates of 65 and 480 sccm, respectively. A glass tube with an inner diameter of 8 cm was attached on the side of the stainless-steel chamber, and a spiral antenna was wound around the glass tube. The antenna was connected to an rf power supply at 13.56 MHz via a matching circuit. Inductively coupled plasmas were produced by applying rf power of 100-160 W to the antenna. We used no specific catalysts in this experiment, and the production of ammonia was obtained by reactions on the stainless-steel chamber wall. Note that the gas-phase production of ammonia is not expected in the low-pressure plasma.

Another vacuum chamber which was equipped with a quadrupole mass spectrometer (QMS) was connected to the plasma chamber via an orifice with a diameter of 0.5 mm, as shown in Fig. 1. The QMS chamber was differentially evacuated using a turbomolecular pump, and the gas in the plasma was sampled into the QMS chamber through the orifice. The orifice was covered with a stainless-steel plate to avoid the transport of positive ions in the plasma to QMS. A difficulty in the detection of ammonia in the $\text{H}_2\text{O}/\text{N}_2$ plasma using QMS was the overlapping of NH_3^+ and OH^+ at $m/z = 17$. OH^+ was produced in QMS by dissociative ionization of H_2O by electron impact ($\text{H}_2\text{O} + e \rightarrow \text{OH}^+ + \text{H} + 2e$). We detected only NH_3^+ by adjusting the energy of electron beam in QMS. Figure 2 shows the relationship between the signal at $m/z = 17$ and the electron energy. The threshold energies of $\text{NH}_3 + e \rightarrow \text{NH}_3^+ + 2e$ and $\text{H}_2\text{O} + e \rightarrow \text{OH}^+ + \text{H} + 2e$ are 10.2 and 18.1 eV, respectively [9, 27]. Hence, the

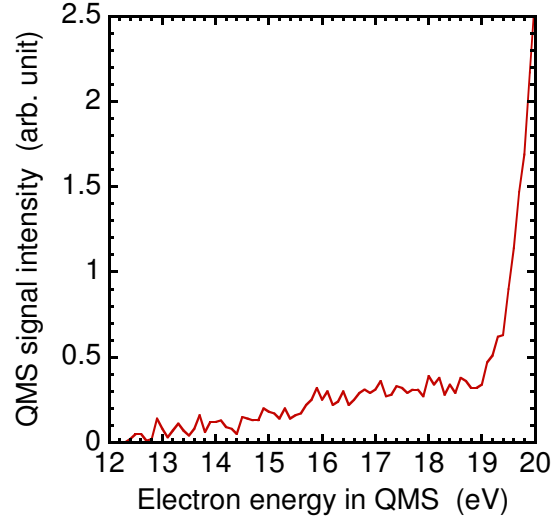


FIGURE 2 Intensity of QMS signal at $m/z = 17$ as a function of electron energy in QMS.

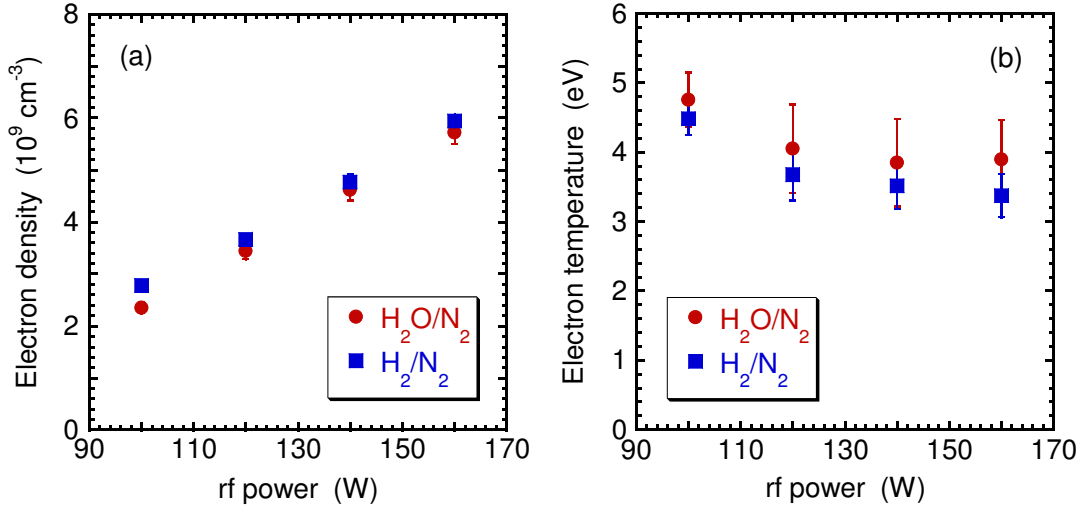


FIGURE 3 (a) Electron density and (b) electron temperature of H₂O/N₂ and H₂/N₂ plasmas as a function of rf power.

step increase in the signal above 18 eV in Fig. 2 is attributed to the detection of OH⁺ produced by $\text{H}_2\text{O} + \text{e} \rightarrow \text{OH}^+ + \text{H} + 2\text{e}$. It was possible to detect only NH₃⁺ by adjusting the electron energy at 15 eV, since the production of OH⁺ was negligible.

3 | RESULTS

The electron densities and the electron temperatures in the H₂O/N₂ and H₂/N₂ plasmas, which were measured using a planar Langmuir probe with a surface area of 45 mm², are shown in Figs. 3(a) and 3(b), respectively. The probe was inserted between the exit of the glass tube and the ion shield. The error bars represent the reproducibility in the multiple measurements (The error bars in Fig. 3(a) are smaller than the sizes of the plot symbols). The electron densities of the plasmas were on the order of 10⁹ cm⁻³. The differences in the electron densities and the electron temperatures of the H₂O/N₂ and H₂/N₂ plasmas were within the magnitudes of the error bars. Because of roughly the same electron densities and the electron temperatures, we can expect the similar rates of electron impact dissociation of N₂ in both the plasmas. The comparison between the H₂O/N₂ and H₂/N₂ plasmas highlights the difference between H₂O and H₂ in the production and destruction processes of ammonia.

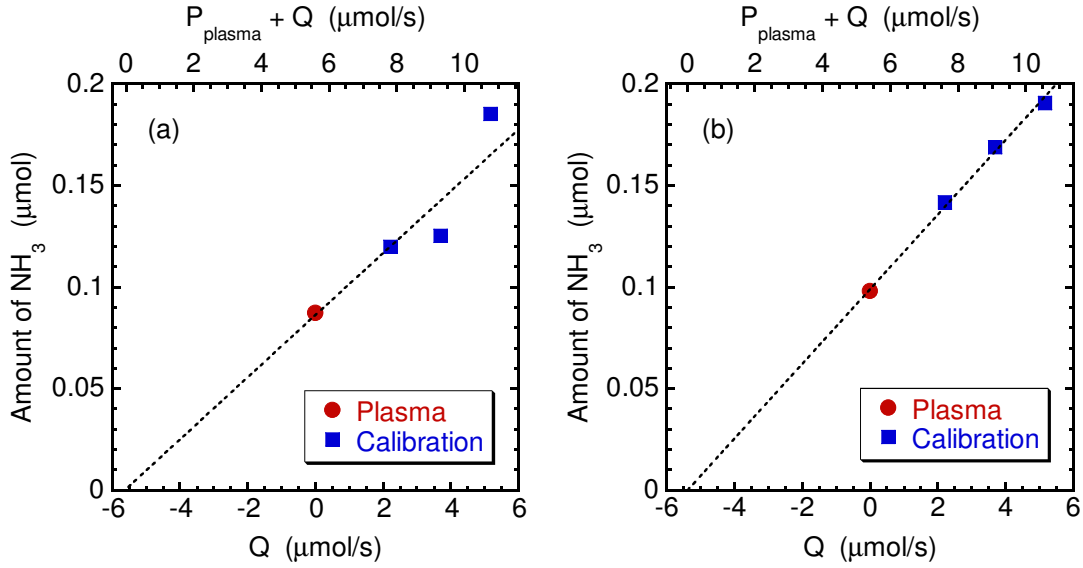


FIGURE 4 Calibration curves for evaluating the production rates and the destruction frequencies of ammonia. The calibration curves show relationships between the amount of ammonia in the plasma chamber and the flow rate of ammonia in (a) H₂O/N₂ and (b) H₂/N₂ plasmas generated at an rf power of 120 W. The amount of ammonia at the origin of the bottom horizontal axis ($Q = 0$) shows that produced by the plasma. The top horizontal axis shows the sum of the production rate in the plasma and the flow rate of ammonia.

When we adjusted the electron energy in QMS at 15 eV, as shown in Fig. 2, the signal at $m/z = 17$ told us the density of ammonia in the plasma chamber. We evaluated the production rate and the destruction frequency of ammonia using the following calibration method. We added a small amount of ammonia (< 10 sccm) from a gas bottle into the plasma. Since the flow rate of ammonia was much lower than the flow rates of nitrogen, water vapor, and hydrogen, we expected that the plasma was not changed by the admixture of ammonia. In fact, we confirmed that the water vapor density or the QMS signal at $m/z = 18$ was not affected by the admixture of ammonia into the H₂O/N₂ plasma. If the plasma was not changed by the admixture of ammonia, the mole number of ammonia in the plasma chamber is given by

$$n(\text{NH}_3) = \frac{1}{\nu_{\text{loss}}}(P_{\text{plasma}} + Q), \quad (1)$$

where P_{plasma} and ν_{loss} are the production rate and the destruction frequency of ammonia, respectively, and Q represents the production rate corresponding to the flow rate of ammonia provided from the gas bottle. P_{plasma} and Q are given in the unit of mol/s. Figure 4 shows the relationships between the amount (the mole number) of ammonia in the plasma region and the flow rate of ammonia. The mole number was obtained by multiplying the density measured using QMS and the capacitance manometer by the volume of the plasma region. The mole number at the origin of the bottom horizontal axis ($Q = 0$) shows the amount of ammonia synthesized by the plasma. The dotted lines shown in Fig. 4 represent the relationship of Eq. (1). Since $n(\text{NH}_3) = 0$ if $P_{\text{plasma}} + Q = 0$, we obtain $P_{\text{plasma}} = 5.6$ and $5.4 \mu\text{mol/s}$ in the H₂O/N₂ and H₂/N₂ plasmas, as shown by the top horizontal axes of Figs. 4(a) and 4(b), respectively. In addition, we can estimate the absolute value of ν_{loss} , which is the sum of the destruction frequency in the plasma (ν_{plasma}) and the pumping loss frequency (ν_{pump}), from the slope of Fig. 4 such that $\nu_{\text{loss}} = 66$ and 55 s^{-1} in the H₂O/N₂ and H₂/N₂ plasmas, respectively. We also evaluated the pumping loss frequency of $\nu_{\text{pump}} = 44 \text{ s}^{-1}$ by carrying out the same calibration procedure without producing a plasma.

The production rates P_{plasma} and the loss frequencies ν_{plasma} observed in the H₂O/N₂ and H₂/N₂ plasmas are plotted in Fig. 5 as a function of the rf power. ν_{plasma} was obtained by subtracting $\nu_{\text{pump}} = 44 \text{ s}^{-1}$ from ν_{loss} evaluated by the calibration procedure. The error bars represent the ambiguity in deducing P_{plasma} and ν_{loss} from calibration curves. As shown in Fig. 5(a), we observed almost the same production rates of ammonia in the H₂O/N₂ and H₂/N₂ plasmas. On the other hand, the destruction frequency of ammonia was 2-4 times higher in the H₂O/N₂ plasma than the H₂/N₂ plasma. The flow rate of hydrogen in the experiment shown in Fig. 5 was 65 sccm, which was lower than the flow rate of water vapor (106 sccm). The lower flow rate of hydrogen was unavoidable to realize the same partial pressure as water vapor. We examined possible ill effects caused by the low flow

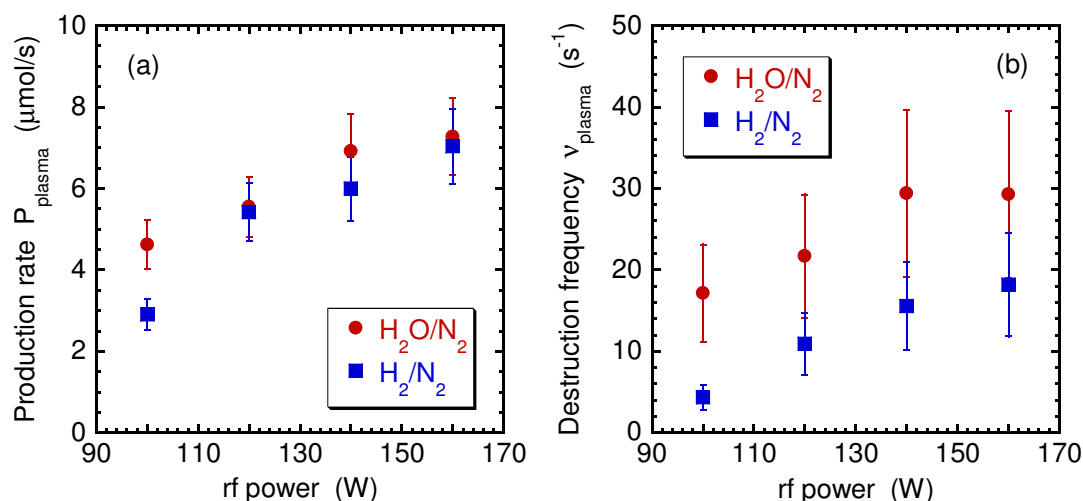


FIGURE 5 Comparisons of (a) production rates and (b) destruction frequencies of ammonia in $\text{H}_2\text{O}/\text{N}_2$ and H_2/N_2 plasmas.

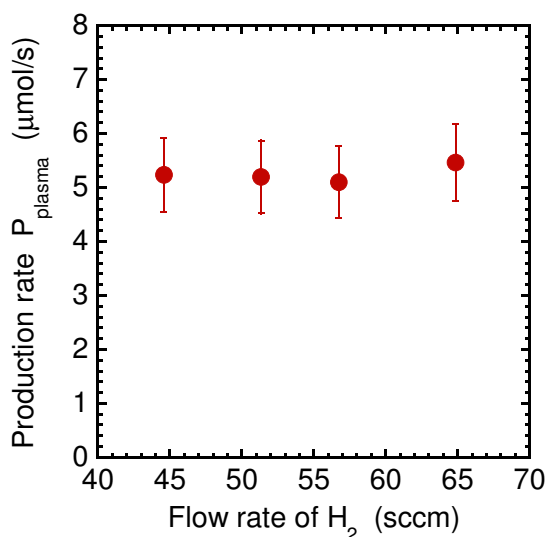


FIGURE 6 Production rate of ammonia in H_2/N_2 plasma as a function of the flow rate of H_2 . The partial pressure of H_2 and the rf power were constant at 2 mTorr and 120 W, respectively.

rate of hydrogen. Since a higher evacuation speed for hydrogen was impossible, we examined the production rate of ammonia at a lower flow rate by reducing the evacuation speed. We used a gate valve at the top of the turbomolecular pump to reduce the evacuation speed. The partial pressures of nitrogen and hydrogen were kept at 10 and 2 mTorr, respectively, and the rf power was constant at 120 W. As a result, as shown in Fig. 6, we observed a constant production rate of ammonia at hydrogen flow rates between 45 and 65 sccm, indicating that the depletion of hydrogen was negligible in the present experimental condition, even though the flow rate of hydrogen was lower than that of water vapor.

4 | DISCUSSION

Almost the same production rates of ammonia shown in Fig. 5(a) suggest that the fluxes of active nitrogen transported to the stainless-steel chamber wall are similar in the $\text{H}_2\text{O}/\text{N}_2$ and H_2/N_2 plasmas. The similar fluxes of active nitrogen are obtained by roughly the same electron density and electron temperature of the $\text{H}_2\text{O}/\text{N}_2$ and H_2/N_2 plasmas shown in Fig. 3. In addition, according to the experimental result, the gas-phase loss of atomic nitrogen in the $\text{H}_2\text{O}/\text{N}_2$ plasma due to $\text{N} + \text{OH} \rightarrow \text{NO} + \text{H}$

is not serious. The rate limiting step is the generation and adsorption of active nitrogen also in the $\text{H}_2\text{O}/\text{N}_2$ plasma, and the fluxes of atomic hydrogen are sufficient for the hydrogenation of nitrogen adsorbed on the stainless-steel chamber wall in both the $\text{H}_2\text{O}/\text{N}_2$ and H_2/N_2 plasmas. H_2O has no negative effects on the production rate of ammonia in the present experimental condition, which is the most important finding obtained by the present work.

The drawback of the $\text{H}_2\text{O}/\text{N}_2$ plasma in comparison with H_2/N_2 is the higher destruction frequency of synthesized ammonia, as shown in Fig. 5(b). Since the mole number of ammonia extracted from the exhaust port of the pumping system per unit time is given by

$$P_{\text{out}} = P_{\text{plasma}} \frac{\nu_{\text{pump}}}{\nu_{\text{pump}} + \nu_{\text{plasma}}}, \quad (2)$$

a higher value of ν_{plasma} results in lower P_{out} . This problem becomes more serious if we use a pumping system with a slower evacuation speed. There are two destruction processes in plasmas: electron impact dissociation and the destruction due to chemical reactions. The frequencies of electron impact dissociation are expected to be similar in the $\text{H}_2\text{O}/\text{N}_2$ and H_2/N_2 plasmas, since the electron density and the electron temperature are roughly the same, as shown in Fig. 3. The electron impact dissociation channel with the lowest threshold energy (3.9 eV) or the highest rate coefficient is $\text{NH}_3 + \text{e} \rightarrow \text{NH} + \text{H}_2 + \text{e}$, which has the maximum cross section of approximately $1 \times 10^{-16} \text{ cm}^2$ at an electron energy around 7 eV [28]. If we calculate the rate coefficient of this dissociation channel on the basis of the cross section data, its frequency amounts to the order of 10 s^{-1} at the electron densities and the electron temperatures shown in Fig. 3. Considering the small rate coefficient of $1.3 \times 10^{-16} \text{ cm}^3/\text{s}$ at 500 K for $\text{NH}_3 + \text{H} \rightarrow \text{NH}_2 + \text{H}_2$ [29], the destruction frequency observed in the H_2/N_2 plasma ($5\text{--}20 \text{ s}^{-1}$) may be explained by electron impact dissociation, and the higher destruction frequency observed in the $\text{H}_2\text{O}/\text{N}_2$ plasma is attributable to additional chemical reactions. We speculate that OH radical, which is produced as the partner of H by electron impact dissociation of H_2O , is the predominant agent in the chemical destruction process of ammonia in the $\text{H}_2\text{O}/\text{N}_2$ plasma. OH is a strong oxidizer. The rate coefficient of $\text{NH}_3 + \text{OH} \rightarrow \text{NH}_2 + \text{H}_2\text{O}$ is $6.6 \times 10^{-13} \text{ cm}^3/\text{s}$ at 500 K [30], which is much higher than the rate coefficient of $\text{NH}_3 + \text{O} \rightarrow \text{NH}_2 + \text{OH}$ ($4.0 \times 10^{-15} \text{ cm}^3/\text{s}$ [31]). The difference in the destruction frequencies in the $\text{H}_2\text{O}/\text{N}_2$ and H_2/N_2 plasmas ($\sim 15 \text{ s}^{-1}$) is explained by an OH density of $\sim 2 \times 10^{13} \text{ cm}^{-3}$. The suppression of the OH density may be a key for the efficient synthesis of ammonia using the $\text{H}_2\text{O}/\text{N}_2$ plasma. If we can find a method to reduce the OH density in the future work, the low-pressure $\text{H}_2\text{O}/\text{N}_2$ plasma will be more attractive as a synthesis process of ammonia without emitting carbon dioxide.

5 | CONCLUSIONS

In this work, we observed almost the same production rates of ammonia in inductively coupled $\text{H}_2\text{O}/\text{N}_2$ and H_2/N_2 plasmas. The partial pressures of H_2O (H_2) and N_2 were 2 and 10 mTorr, respectively. The production rate of $4\text{--}7 \mu\text{mol/s}$ was obtained at an rf power of 100–160 W, corresponding to an energy cost of 23–25 MJ/mol. Note that the rf power of 100–160 W was not the power absorbed by the plasma but the power yielded from the power supply. The energy cost may be improved if we can use a modified rf system, since the power consumed for heating the matching circuit and the transmission line is not negligible in the present experimental condition [32, 33]. On the other hand, the destruction frequency of ammonia in the $\text{H}_2\text{O}/\text{N}_2$ plasma was 2–4 times higher than that in the H_2/N_2 plasma. The higher destruction frequency in the $\text{H}_2\text{O}/\text{N}_2$ plasma is considered to be due to $\text{NH}_3 + \text{OH} \rightarrow \text{NH}_2 + \text{H}_2\text{O}$. Considering the expensive production cost of “green hydrogen”, the use of water vapor has great advantage as the hydrogen source in the ammonia synthesis. If we can find a method to reduce the destruction frequency of ammonia by reducing the OH density in the $\text{H}_2\text{O}/\text{N}_2$ plasma, the $\text{H}_2\text{O}/\text{N}_2$ plasma will be more attractive as a synthesis process of ammonia without emitting carbon dioxide.

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DARA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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