

Application of Microplasma for NO_x Removal

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Abstract—In this paper, microplasma is investigated, which occurs between the narrow gap of the electrodes covered with dielectric materials. The discharge gap is set on the order of micrometers by changing a spacer from 0 to 100 μm . In this paper, the characteristics of microplasma, such as discharge voltages, discharge currents, and discharge power that is obtained with the help of Lissajous figures, and the relationships between these electric characteristics are presented. The characteristics of ozone generation by a microplasma electrode are investigated. Treatment by microplasma for the simulated exhaust gas, which contains NO_x and C₃H₈ as a role of HC and CO, is estimated experimentally. In the absence of O₂, the exhaust gas is decomposed by nitrogen atomic species in particular. In the presence of O₂, it is decomposed by nitrogen atomic species and oxidized by ozone. By-product analysis of treated gases is carried out by Fourier transform infrared spectroscopy and gas chromatography–mass spectrometry. When HC, NO_x, and N₂ are included in the simulated exhaust gas, HCN, N₂O, and CH₄ are confirmed as by-products of the microplasma treatment.

Index Terms—Air pollution control, exhaust gas, microplasma, NO_x, ozone.

I. INTRODUCTION

NON-THERMAL plasma is widely used for environmental protection and plasma-enhanced chemical synthesis or reaction, particularly ozone generation [1]–[4] and polluted gas control such as dissociations of NO_x and HC with a dielectric barrier discharge [5]–[12] and a pulsed corona [13]–[15].

Microplasma could be generated at lower discharge voltages than those discharge methods because of its narrow gaps. Therefore, the size of the power source of microplasma could be smaller than that of a corona discharge and an atmospheric glow discharge.

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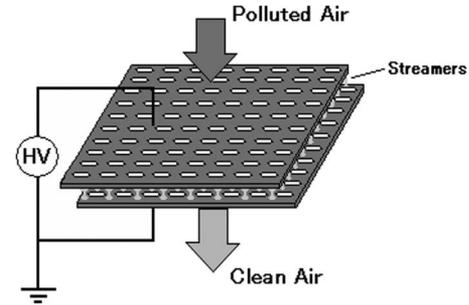


Fig. 1. Schematic image of microplasma electrodes. The pressure loss between the electrodes is very small.

This point would be significant for indoor-air or exhaust-gas treatment, particularly for small cars or motorcycles. We have investigated the feasibility of microplasma for removing the simulated exhaust gas of gasoline engines. Microplasma could be generated at a discharge gap of less than 100 μm , particularly 10 μm with only around 1 kV.

Recently, the characteristics of microplasma have been investigated [16]. There are few applications for environmental protection such as formaldehyde removal of indoor air [17], [18].

II. ABOUT MICROPLASMA

Fig. 1 shows a schematic image of the electrodes used in the experiments. It is a stainless steel covered with dielectric barrier materials in both faces and is 120 cm² in area. The thickness of the dielectric material is 100 μm (Fig. 2). There are many holes in the electrode with \varnothing 1.5 mm that results in an aperture ratio of 25%.

The gas is flowed through the holes and reacted with electrons between the electrodes, which are supplied with a self-made high-voltage ac power. The pressure loss is about 30-mmH₂O in a gas flow rate of 8.5 L/min. The pressure loss is very small, and this reactor could be applied to exhaust-gas treatments with large gas flows.

In this experiment, the discharge gap between the electrodes is set to less than 100 μm to realize the minimum sparking voltage at atmospheric pressure based on Pachen's law. Table I shows the minimum sparking voltage and the minimum gaps for air, nitrogen, and oxygen.

Fig. 3 shows a schematic image of the removal process of microplasma between the electrodes. The simulated exhaust gas contains NO_x, and HC and CO could be decomposed by atomic oxygen and the nitrogen species generated between the electrodes.

Due to the assumed value of the dielectric constant of the dielectric material $\epsilon_{r1} = 10^4$ and according to

$$D = \epsilon_1 E_1 = \epsilon_2 E_2 \quad (1)$$

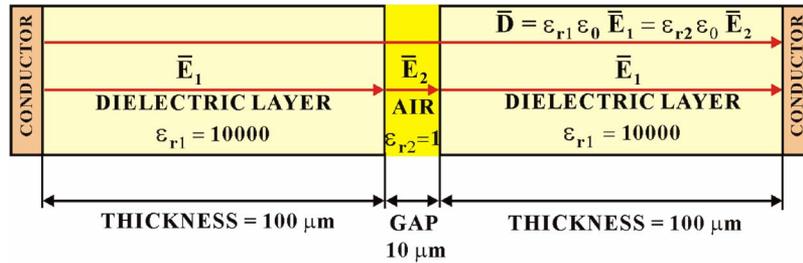


Fig. 2. Electric field inside the capacitor formed by a pair of electrodes consisted in two dielectric layers and the air gap. Due to the high dielectric constant of the dielectric layer $\epsilon_{r1} = 10^4$, a high-intensity electric field in the air gap $E_2 = 10^7 - 10^8$ V/m is obtained.

TABLE I
MINIMUM SPARKING VOLTAGE AND DISCHARGE GAP
FOR VARIOUS GASES

Gas	minimum sparking voltage [V]	minimum gap [μm]
Air	330	7.5
Oxygen	450	9.2
Nitrogen	275	9.9

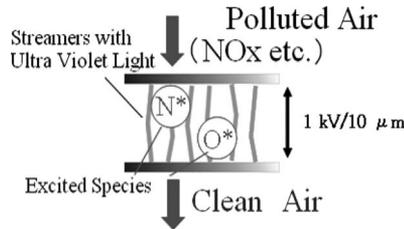


Fig. 3. Schematic image of the removal process of microplasma between the electrodes.

where D is the electric displacement field, E_1 and E_2 are the intensities of the electric field in the dielectric layer and in the air gap, respectively, and $\epsilon_1 = \epsilon_{r1}\epsilon_0$ and $\epsilon_2 = \epsilon_{r2}\epsilon_0$ are the permittivities, respectively, of the dielectric layer and of the air, where ϵ_0 is the vacuum permittivity, it results in

$$E_2 = \frac{\epsilon_{r1}\epsilon_0}{\epsilon_{r2}\epsilon_0} E_1 = 10^4 E_1. \quad (2)$$

At a gap between the electrodes of $10 \mu\text{m}$ and at a thickness of the dielectric barrier of $100 \mu\text{m}$, and according to (2), a high electrical field ($E_2 = 10^7 - 10^8$ V/m) is obtained, which assures the formation of nonthermal plasma. High-energy electrons and active species are generated in such a microplasma, which is activated by high electrical field.

III. EXPERIMENTAL SETUP

A. Analysis of Gas Composition

Fig. 4 shows the experimental setup. The gas mixture is produced by using gas cylinders of N_2 , NO , O_2 , and C_3H_8 in order to simulate the exhaust gases from the gasoline engines at high-load conditions. Concentrations of NO_x and O_3 are measured by a chemiluminescence NO_x analyzer (Shimadzu, NOA-7000) and an ultraviolet-absorption-type ozone monitor (Ebara, EG-2001B). Concentrations of CO and HC are also measured by a CO/HC analyzer with nondispersive infrared analyzer (Horiba, MEXA-324G).

The by-products, such as HC , NO_x , CO , and CO_2 , generated by a microplasma are analyzed by Fourier transform infrared spectroscopy (FTIR; Shimadzu, IRPrestige-21) and gas chromatography–mass spectrometry (Agilent, 5972 and 5890).

B. Power Source for a Microplasma

A self-made high-frequency (15–20 kHz) power source is assembled to generate a high-voltage range of around 1 kV. Due to the relatively low voltages and very small gaps necessary for generating microplasma, only a downsized power supply is needed. The high voltage supplied to the electrode and its corresponding discharge currents are measured by a high-voltage divider (Tektronix, P6015A), an ac current probe (Tektronix, P6022), and a digital oscilloscope (Tektronix, TDS 2014). Discharge power is estimated by using a Lissajous figure.

IV. CHARACTERISTICS OF MICROPLASMA

A. Discharge Voltage of Microplasma

Fig. 5 shows sample waveforms of microplasma discharge voltage and discharge current. The applied voltage is a high-frequency ac. Fig. 5 shows the discharge voltage of 1.2 kV, which is applied to the microplasma electrode, and the corresponding discharge current of 40 mA, which contains capacitive currents. The spike currents that appeared due to the microdischarges are convoluted on the current waveform and were confirmed at the steepest slopes of the discharge voltage.

Discharge power is estimated by using a Lissajous figure, as shown in Fig. 6, and by measuring the capacitance charge, which is connected between the electrode and ground.

B. Discharge Power of Microplasma

Fig. 7 shows the characteristics of the discharge power and the discharge voltage of microplasma in air. In order to set the discharge gaps, a spacer is used. In this figure, a discharge gap of $0 \mu\text{m}$ stands for the electrodes put together without any spacer. In this case, microplasma could be generated on each surface of the electrode, particularly at the circumference of the edge of each hole.

Microplasma could be generated on each surface of the electrode, particularly at the circumference of the edge of each hole. The discharge form of a microplasma electrode could be a combination of a surface discharge and a barrier discharge. Due to this reason, the corona onset voltages for 10- and $0\text{-}\mu\text{m}$ gaps

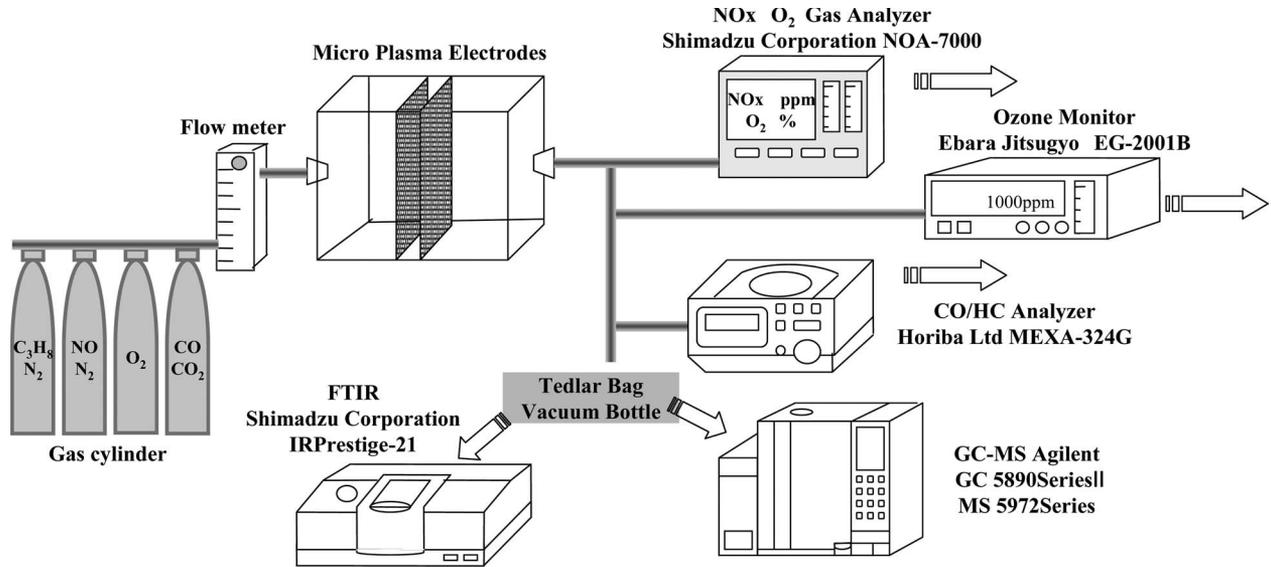


Fig. 4. Experimental setup including electrical measurements and analysis of gas composition.

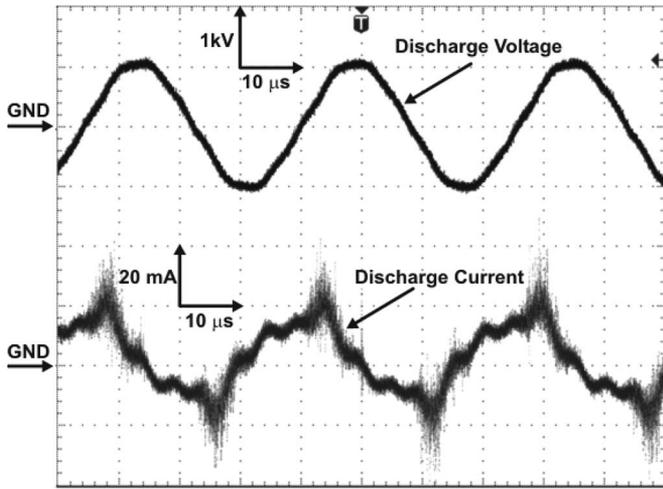


Fig. 5. Sample waveform of a discharge voltage and a discharge current.

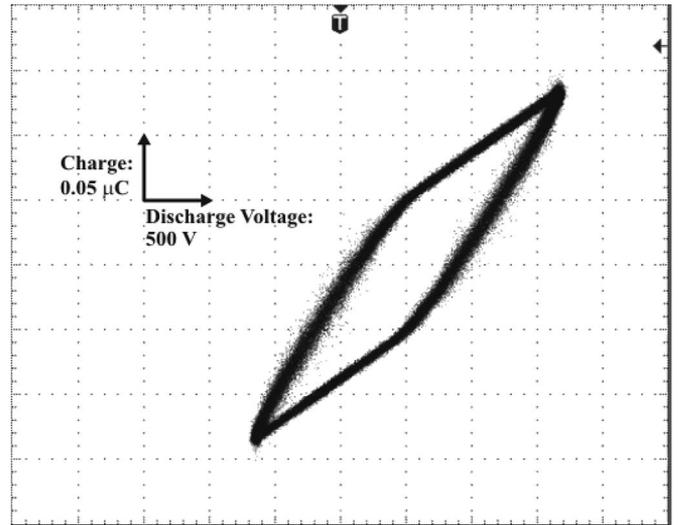


Fig. 6. Example of a Lissajous figure.

tend to be similar. The characteristics of the discharge power also tend to be similar for the discharge gaps of 0 and 10 μm.

The sparking voltage becomes lower when the discharge gap is smaller. For example, at a discharge gap of 10 μm, discharge occurs at 0.8 kV; on the contrary, in the case of 100 μm, it occurs at 1.2 kV.

When the discharge starts, the area of the discharge was only a part of the electrode. As the discharge voltage is raised, the area spreads. Therefore, the discharge power is small when the discharge starts. With smaller discharge gap, microplasma could be generated with smaller discharge power.

V. GAS-REMOVAL EXPERIMENT

A. NO_x Removal

Treatment of air pollutants is one of the important issues regarding global environmental protection. In this paper, NO_x, which is contained in the exhaust gas, is the target to control for one example of the treatment of microplasma.

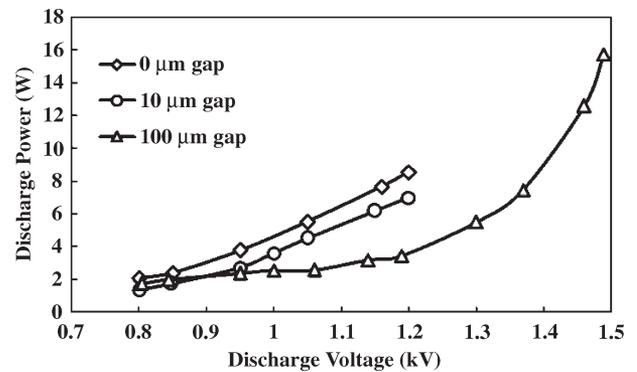


Fig. 7. Characteristics of the discharge voltage versus the discharge power at various discharge gaps.

Fig. 8 shows the NO_x removal process for the gas composition containing 100-ppm NO, 0-ppm C₃H₈, and N₂ balance and for the gas composition containing 100-ppm NO, 500-ppm C₃H₈, and N₂ balance (Table II). The objective of the

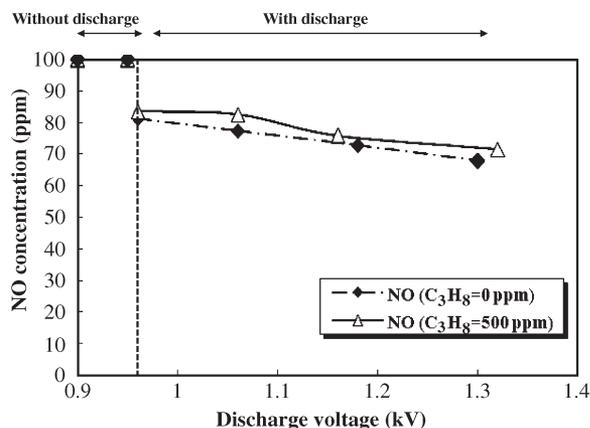


Fig. 8. Removal of NO by microplasma at a gas flow rate of 5 L/min.

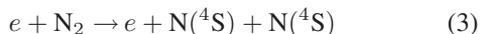
TABLE II
GAS COMPOSITION OF FIG. 8

Experimental conditions	NO (ppm)	C ₃ H ₈ (ppm)	N ₂
<1> ♦	100	0	Balance
<2> △	100	500	Balance

experiments was to analyze the NO_x removal process in the presence and in the absence of C₃H₈.

The experiments were carried out at a gas flow rate of 5 L/min, a discharge gap of 10 μm, and a temperature of the gas of 25 °C.

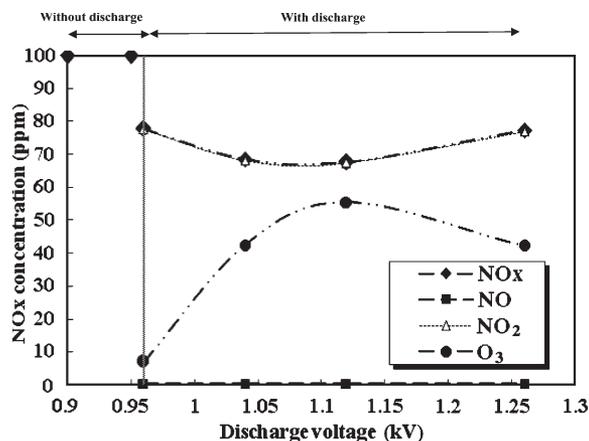
The gas composition without C₃H₈ was treated by a microplasma. As the discharge voltage increases, the NO_x concentration decreases from 80 ppm at the beginning of the discharge process, corresponding to a discharge voltage of 0.96 kV, to 72 ppm, corresponding to a discharge voltage of 1.3 kV. NO could be chemically reduced to N₂ by atomic nitrogen species, produced by electron impact of N₂ [8]



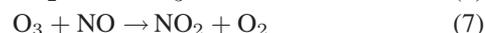
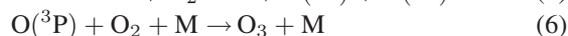
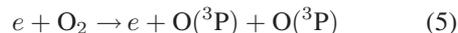
N(^4S) is a ground-state nitrogen atom. The electron-impact dissociation of NO is few because the concentration of N₂ is much higher than that of NO, and most of the high-energy electrons react with N₂.

With the increase of the discharge voltage, the concentration of NO_x in the gas composition with C₃H₈ treated by a microplasma decreases from an initial concentration of 82 ppm corresponding to a discharge voltage of 0.96 kV to 78 ppm corresponding to a discharge voltage of 1.33 kV. C₃H₈ does not play a role in the dissociation of NO, since the effect of atomic nitrogen species is much larger. In this experiment, NO concentration is almost equal to NO_x concentration.

Fig. 9 shows the removal of NO_x by a microplasma at a discharge gap of 10 μm at 25 °C. The gas composition was 100-ppm NO, 5% O₂, and N₂ balance, and it was treated by a microplasma at a gas flow rate of 5 L/min.

Fig. 9. Removal of NO by microplasma at a gas flow of 5 L/min. Gas composition: 100-ppm NO, 5% O₂, and N₂ balance.

With small amounts of O₂, NO could chemically be oxidized to NO₂ by O₃, which is produced by electron impact of O₂ [8]



O(^3P) is a ground-state oxygen atom, and M is either N₂ or O₂. Microplasma could be generated at a discharge voltage of about 1 kV. At a discharge voltage of 0.96 kV, the concentration of NO_x was decreased to 78 from 100 ppm. The removed quantity of NO_x increased proportionally to the discharge voltages, and the quantity of ozone increases. It has a certain peak at a discharge voltage of about 1.1 kV. This could be explained by the reaction of ozone with NO and also with NO₂ according to (7) and (8) [19]. When the discharge voltage exceeds 1.1 kV, the concentration of NO_x increases, and the quantity of ozone decreases. By increasing the discharge voltage, more atomic nitrogen species are produced, which reacts with ozone, resulting with a newly formed NO_x. NO_x is generated by the following processes [1], [8]: (5), (6), (7), and

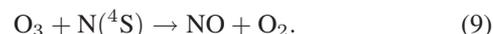


Fig. 10 shows the removal of NO_x by a microplasma at a discharge gap of 10 μm at 25 °C. The gas composition was 100-ppm NO, 5% O₂, 500-ppm C₃H₈, and N₂ balance.

With small amounts of C₃H₈, the concentration of NO_x is increased by a microplasma, and the oxidation of NO to NO₂ decreased. Almost no O₃ could be detected in this experimental condition. Most of the generated O₃ is reacted to other materials such as NO, C₃H₈, and, in particular, nitrogen atoms to be NO that is already shown in (9).

B. By-Product Analysis With FTIR

By-products must be found after the treatment of microplasma, particularly gas-phase reactions in air or exhaust gas containing various pollutants such as hydrocarbons, which generate hazardous pollutants again.

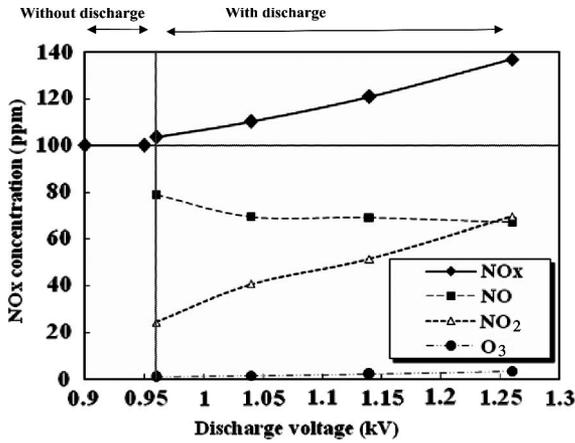


Fig. 10. Removal of NO by microplasma at a gas flow rate of 5 L/min. Gas composition: 100-ppm NO, 5% O₂, 500-ppm C₃H₈, and N₂ balance.

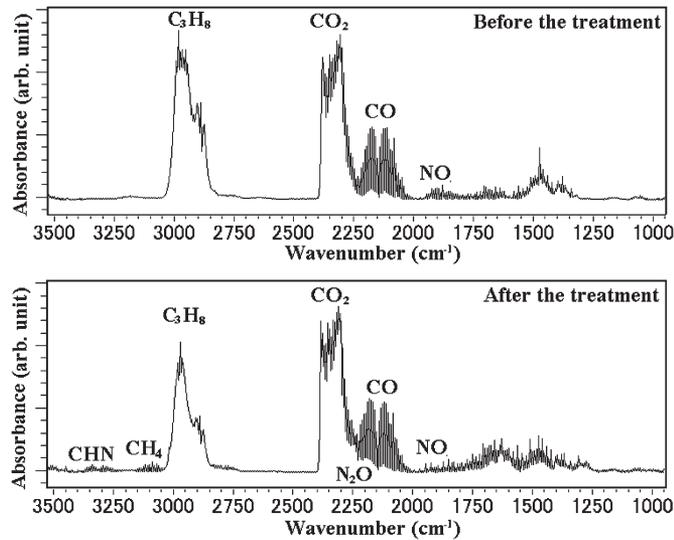


Fig. 11. By-product analysis obtained by FTIR in the absence of O₂. Gas composition: 4.02% CO, 3510-ppm C₃H₈, 1215-ppm NO, 12.02% CO₂, and N₂ balance. Discharge voltage: 1.3 kV. Discharge gap: 10 μm. Gas flow rate: 0.5 L/min.

In the case of treating NO_x with ethylene and oxygen as a simulated gas, acetaldehydes and acetic acids are found as by-products after the plasma treatment [20]. Methanol is a by-product material as a result of the conversion of methane [21], [22].

In this experiment, an FTIR and a 10-m gas cell were used to identify the final by-products of treating the simulated exhaust gases by a microplasma at a discharge gap of 10 μm. The gas cylinder adjusted the simulated gas composition of 4.02% CO, 3510-ppm C₃H₈, 1215-ppm NO, 12.02% CO₂, and N₂ balance at a gas flow rate of 0.5 L/min to increase the gas retention time between the electrodes.

Fig. 11 shows the spectra of by-product analysis obtained by FTIR in the absence of O₂. The experimental conditions of Fig. 10 are at a discharge voltage of 1.3 kV and a discharge gap of 10 μm. The upper figure shows the spectrum of before treatment with microplasma, and the lower figure shows that of after the treatment. Table III shows the changes of the concentration of simulated gases by a microplasma in the absence of

TABLE III
CONCENTRATION CHANGE OF SIMULATED GASES
IN THE ABSENCE OF OXYGEN

	C ₃ H ₈ <3510 ppm>	CO <4.02%>	NO{NO ₂ } <1215 ppm>
Before discharge	3362 (3331)	4.04 (4.19)	972 {32}
After discharge	1233 (1457)	3.97 (4.02)	492 {20}

<> shows a calculated concentration of C₃H₈, CO and NO_x.

() shows a measurement concentration of C₃H₈ and CO with CO/HC analyzer.

{ } shows a concentration of NO₂.

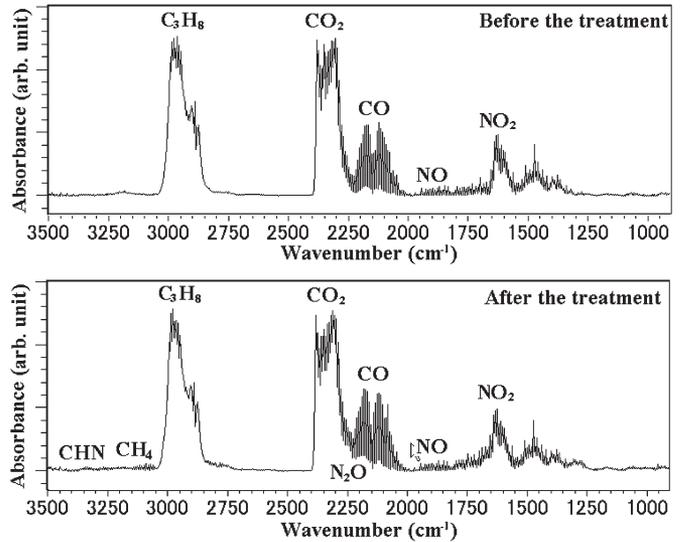


Fig. 12. By-product analysis obtained by FTIR in the existence of O₂. Gas composition: 3.82% CO, 3335-ppm C₃H₈, 1154-ppm NO, 11.42% CO₂, 5% O₂, and N₂ balance. Discharge voltage: 1.3 kV. Discharge gap: 10 μm. Gas flow rate: 0.5 L/min.

O₂. The concentration of NO_x is measured by a NO_x analyzer. The concentrations of C₃H₈ and CO are cross-checked by using an FTIR analyzer and a CO/HC analyzer.

As shown in Fig. 11 and Table III, HCN, CH₄, and N₂O are confirmed as by-products, and C₃H₈, CO, and NO are decomposed by a microplasma after the discharge. According to Table III, the amount of C₃H₈ decreases in the absence of O₂, resulting in the formation of HCN and CH₄.

Fig. 12 shows the spectra of by-product analysis obtained by FTIR with oxygen concentration of 5%.

The experimental conditions of Fig. 11 are at a discharge voltage of 1.3 kV and a discharge gap of 10 μm. The gas composition of the simulated gas is adjusted to 3.82% CO, 3335-ppm C₃H₈, 1154-ppm NO, 11.42% CO₂, 5% O₂, and N₂ balance. The upper figure shows the spectrum of before the treatment with microplasma, and the lower figure shows that of after the treatment.

Table IV shows the changes of the concentration of simulated gas by a microplasma with oxygen concentration of 5%. These are estimated from the FTIR spectra and by using a NO_x analyzer and a CO/HC analyzer.

As shown in Fig. 12 and Table IV, HCN, CH₄, CO, NO₂, and N₂O are generated as by-products, and C₃H₈ and NO are decomposed by a microplasma after the discharge. According

TABLE IV
CONCENTRATION CHANGE OF SIMULATED GASES WITH 5% OXYGEN

	C ₃ H ₈ <3335 ppm>	CO <3.82%>	NO {NO ₂ } <1154 ppm>
Before discharge	3198 (3281)	3.95 (4.1)	940 {145}
after discharge	2826 (2974)	4.62 (4.79)	873 {313}

<> shows a calculated concentration of C₃H₈, CO and NO_x.

() shows a measurement concentration of C₃H₈ and CO with CO/HC analyzer.

{ } shows a concentration of NO₂.

to Table IV, the amount of C₃H₈ decreases in the presence of O₂, with C reacting with O, and it results in the formation of CO and also of HCN and CH₄.

C. Energy Efficiency for Gas Treatment

The specific input energy (SIE) is defined as the total discharge power dissipated per unit volume of the gas [23]

$$\text{SIE [J/L]} = \frac{\text{Electrical Input Power [J/s]}}{\text{Gas Flow Rate [L/s]}} \quad (10)$$

Fig. 7 shows the characteristics of the discharge power and the discharge voltage of microplasma in air. For a discharge gap of 10 μm and a discharge voltage of 1.2 kV, a corresponding electrical input power of 7 W was measured. At a gas flow rate of 5 L/min, according to (10)

$$\text{SIE}_{(1.2 \text{ kV and } 10\text{-}\mu\text{m gap})} = 7/0.0833 = 84 \text{ J/L} \quad (11)$$

The measurements of the discharge power and discharge voltage shown in Fig. 7 were performed for one pair of electrodes. The measured power contains also the losses in the power-supply circuit. Two pairs of electrodes were used in order to measure the discharge power. At a discharge voltage of 1.2 kV and a discharge gap of 10 μm, a discharge power of 9.5 W was measured. By subtracting the value of the discharge power shown in Fig. 7 for the discharge voltage of 1.2 kV, the 2.5-W value of the discharge power for one pair of electrodes is obtained. Thus, according to (10), the SIE for one pair of electrodes is

$$\text{SIE}_{(1.2 \text{ kV and } 10\text{-}\mu\text{m gap})} = 2.5/0.0833 = 30 \text{ J/L} \quad (12)$$

According to Fig. 8, for the gas composition of 100-ppm NO and N₂ balance, the NO removal concentration at the discharge voltage of 1.2 kV was 28 ppm. The energy efficiency at this discharge voltage was 4.5 g (NO)/kW · h. Although the energy efficiency of the microplasma reactor is lower than that of the conventional plasma reactor [24], [25], due to the small sizes of the reactor and power supplies, microplasma is a technology that can be applied at mobile sources such as small automobiles and motorcycles.

VI. CONCLUSION

We have investigated treatment and removal of simulated gas by a microplasma. Removal of NO_x and HC by a microplasma

is considered to be effective. The following are the conclusions drawn from a series of experiments.

- 1) Microplasma is generated around 1 kV at a discharge gap of 10 μm.
- 2) When the simulated gas, including NO, O₂, and N₂, is treated by a microplasma, the value of NO_x removal is maximum at a certain discharge voltage.
- 3) When the simulated gas, including NO, C₃H₈, O₂, and N₂, is treated by a microplasma, the concentration of NO_x increases, and the oxidation of NO to NO₂ decreases. This phenomenon could be concerned with C₃H₈.
- 4) The oxygen and nitrogen atomic species generated by a microplasma could have oxidized NO to NO₂ and decomposed NO and C₃H₈. However, on simulated gas composition, CHN, CH₄, N₂O, and CO are confirmed as by-products.

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