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Abatement of perfluorocarbons with combined plasma catalysis in atmospheric-pressure environment

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Abstract

Perfluorocarbons (PFCs) are widely used in semiconductor industry. However, they intensely absorb infrared radiation and consequently aggravate the greenhouse effect. A combined plasma catalysis (CPC) technology is developed to destroy PFCs. For practical purpose the CPC is designed to function at room temperature and atmospheric pressure. In addition, this study compares three kinds of operations, i.e. catalysis, plasmas and CPC. Results indicate that CPC achieves the highest PFC destruction efficiency. Complete destructions are achieved for SF_6 and NF_3. Removal efficiencies for CF_4 and C_2F_6 are 66 and 83%, respectively. The products formed in CPC process for CF_4 and C_2F_6 are mainly CO_2 and a little CO. In contrast, the products formed in plasma process include CO_2, CO, COF_2, and CF_4. In brief, this study demonstrates that CPC is of a higher efficiency and less-toxic products for PFC removal compared with plasma technology at the given conditions.

Keywords: Perfluorocarbons (PFCs); Combined plasma catalysis (CPC); Air pollution control technology; Global warming

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1. Introduction

Along with the rapid growth and high profits of semiconductor industries nowadays, various kinds of air pollutants are also inevitably generated and possibly released from semiconductor manufacturing processes. As the manufacturing processes becomes more sophisticated for making larger wafers, the chemicals used and the pollution associated also become more complicated. Gaseous pollutants emitted from semiconductor manufacturing processes can be generally classified as volatile organic compounds (VOCs), acid gases and perfluorocompounds (PFCs). PFCs are potential contributors to the global warming, which is adverse to the global environment and has caused much public concern.

PFCs including CF_4, C_2F_6, C_3F_8, CHF_3, SF_6, and NF_3 are extensively used as etching and cleaning gases in semiconductor manufacturing processes. These PFCs are chemically inert and noncorrosive gases that intensely absorb infrared radiation with relatively long lifetimes in the atmosphere. Therefore, once released into the atmosphere they would aggravate the earth's greenhouse effect with relatively high global warming potentials (GWPs) compared to CO_2. The atmospheric lifetimes and GWP_{100} (global warming potential with time horizon of 100 years) of PFCs are greatly larger than that of traditional greenhouse gases such as CO_2, CH_4, and N_2O (see Table 1).

In the semiconductor manufacturing processes, the exhausted gases can be treated either before or after the vacuum pump; namely be treated at either low pressure or atmospheric pressure. The major disadvantage of implementing low-pressure abatement (i.e., treatment upstream of the vacuum pump) is possible damage or malfunction of the vacuum pumps due to acid formation and particle accumulation. To minimize the corrosion to vacuum pumps, the better way is to treat exhausted gases downstream the vacuum pump. Technologies available for abating PFC emissions from semiconductor industries include combustion, catalytic oxidation, and plasma processing.

Combustion is the most developed technology for PFC control on the end-of-pipe treatment basis. A combustor equipped with natural gas or hydrogen injection system has been demonstrated a greater than 90% abatement efficiency for C_2F_6, NF_3 and SF_6, but poor for CF_4 abatement possibly due to its high stability [1]. The cost associated with fuels and solution needed for scrubbing by-products such as HF is expensive. In addition, it has the side effects of generating NO_x and incomplete combustion products.

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Table 1
Atmospheric lifetimes and GWP_{100} of greenhouse gases [18]

Greenhouse gases	Atmospheric lifetime (year)	GWP_{100}
CO_2	50–200	1
CF_4	50000	6500
C_2F_6	10000	9200
SF_6	3200	23900
C_3F_8	2600–7000	7000
CHF_3	250–390	11700
C_4F_8	3200	8700
CH_4	12	21
N_2O	120	310
NF_3	50–740	8000

Catalytic oxidation offers a low-cost alternative compared with combustion technology. It is also proposed as a technology for destroying low concentrations (<1000 ppm) of halogen-containing organic compounds in air [2]. The effectiveness of destroying chlorine-containing organic molecules with platinum group metals on an aluminum oxide support has been demonstrated without significant loss of reactivity [2,3]. However, catalysts developed to oxidize fluorine-containing organic molecules are less successful due to deactivation [4–8]. It is possibly attributed to three causes, i.e.: (1) platinum group metals can form volatile halide compounds and are not tolerant of halogens at temperature >400 °C [5]; (2) aluminum oxide transforms to AlF_3 [5]; and (3) presence of SiF_4 in the gas streams poisons the catalysts, necessitating the feed flow be scrubbed upstream the catalyst bed [9].

Plasma technology has the advantages of high electron temperature which is of great potential to PFC abatement. Nonthermal plasma technologies (NTPs) including surface wave plasma, arc power plasma, microwave plasma, dielectric barrier discharges (DBDs) have been investigated in the last decade as alternate technologies for controlling PFC emissions [10–15]. However, the by-products from NTPs are complex and sometimes parts of them are even noxious to human health or harmful to the environment. In recent years, the concept of combining catalysis with plasma has attracted great research interests worldwide.

Destruction of gaseous pollutants in NTP is mainly attributed to two elements, i.e. electrons and radicals. The electrons are generated by high electric field strength which is either formed by high voltage or induced by high frequency. For DBDs, the driven voltage is typically in the range of several kilovolts to tens of kilovolts. On the other hand, the frequency is typically within 50–20,000 Hz. The mean electron energy and electron density are 2–5 eV and 10^{12}–10^{15} cm^{-3}, respectively. Such electron energy is capable of decomposing toxic gases. For instance, typical bond energy of chemicals is ca. 3–5 eV (N–F, C–C, S–F, and C–F are 2.89, 3.59, 4, and 5.04 eV, respectively). In addition to electrons, radicals are intensively generated by electron-impact dissociation. O and OH are the most important radicals that generally form in electron-impact dissociation with O_2 and $H_2O_{(g)}$, respectively. Reaction rate constants of O and OH are essentially high for organic chemicals; however, very low for PFCs. The critical pathway for PFC decomposition relies on electron-impact reactions. Therefore, increase of electron energy and electron density favors PFC abatement by NTPs.

In plasma chemistry of decomposing air pollutants, the first step is generally through electron-impact reactions. For instance, one major channel for C_2F_6 decomposition is through electron-impact dissociations:

$$e + C_2F_6 \rightarrow CF_3 + CF_3 + e \tag{1}$$

$$e + C_2F_6 \rightarrow C_2F_5 + F + e \tag{2}$$

Another channel for C_2F_6 decomposition is through the dissociative recombination of ${C_2F_6}^+$ ions with electrons:

$$e + C_2F_6 \rightarrow {C_2F_6}^+ + 2e \tag{3}$$

$$e + {C_2F_6}^+ \rightarrow CF_3 + CF_3 \tag{4}$$

In the meantime, recombinations of gas-phase radicals (F and CF_3) formed by plasmas take place:

$$CF_3 + CF_3 \rightarrow C_2F_6 \tag{5}$$

$$C_2F_5 + F \rightarrow C_2F_6 \tag{6}$$

$$CF_3 + F \rightarrow CF_4 \tag{7}$$

It is noticeable that reaction rates of radical recombinations are essentially fast. In addition, CF_4 is also a greenhouse gas (see Table 1) and needs further treatment. Therefore, regeneration of C_2F_6 and CF_4 needs further consideration because their formations may greatly reduce the overall abatement efficiency. To avoid this disadvantage, existence of active species which can rapidly react with PFCs' fragments in the gas streams is a reasonable approach. O_2, H_2, $H_2O_{(g)}$ and hydrocarbons are good candidates for these reactions. As they are collided by energetic electrons, O, H, OH and CH_i are formed and react with PFC fragments speedily. These active radicals not only prevent the regeneration of PFCs but also react with the fragments to form final products. In other words, destruction efficiency of plasma technology can be enhanced by inhibiting recombination of PFC fragments.

The objective of consuming F and CF_i radicals can also be achieved with catalysis. Fluorine atoms can be lost on surfaces by adsorption followed by recombination [16], e.g.,

$$F:S + F:S \rightarrow F_{2(g)} + 2S \tag{8}$$

The behavior of CF_3 radicals on catalyst surfaces is complicated. On nonactive surfaces, CF_3 radicals can be adsorbed without dissociation and desorbs as CF_3 radicals. However, on active surfaces, CF_3 radicals are generally dissociatively adsorbed, producing a carbon atom and three fluorine atoms that each bonds to the active site. In addition, CF_3 radicals can be oxidized by oxygen atoms (and, possibly, O_2 molecules) on the surfaces [16]. On the other hand, gas-phase oxygen atoms abundantly generated by plasmas are beneficial to activation of n-type catalysts [17]. Based

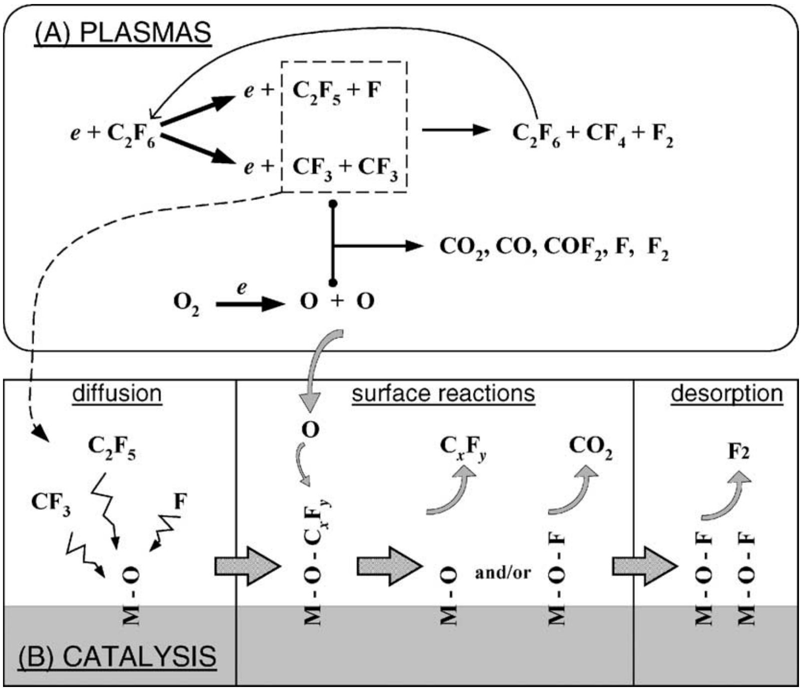

Fig. 1. Possible pathways of C_2F_6 abatement by CPC. The top one shows the major pathways of processing C_2F_6 by plasmas (A); while the bottom one by catalysis (B).

on previous discussions, a possible simple pathway for C_2F_6 abatement by a technology integrating plasmas with catalysis is illustrated in Fig. 1. As described, combining catalysis with plasmas could possibly enhance the consuming rate of free radicals and prevent recombinations of C_2F_6 and CF_4, resulting in a higher overall efficiency compared with each individual process.

This study is motivated to develop an innovative technology named combined plasma catalysis (CPC) which integrates two important features including high selectivity of catalysis technology and high efficiency of plasma technology. CPC consists of a DBD reactor and catalysts. CPC is designed to operate at room temperature and under atmospheric pressure, which means without impacting the semiconductor manufacturing process and with an energy-saving feature. This study aims to investigate the performance of PFC abatement with three operation modes, i.e. catalysis only, plasmas only, and CPC. The end-products in processing PFCs are discussed as well.

2. Experimental

Fig. 2 shows the laboratory-scale experimental system designed and constructed to evaluate the effectiveness of DBD and CPC for PFC abatement. The simulated gas stream was provided by compressed gas cylinders including N_2, O_2, PFCs, and Ar. A set of mass flow meters (Teledyne Hastings-Raydist, HFC 202) were used to regulate the flow rates of feeding gases. Composition and flow rate of the gas streams could then be accurately controlled. The gas streams of all tests were kept at a gas flow rate of 600 standard-cubic-centimeter-per-minute (sccm) and under atmospheric pressure. The objective of this study is to develop a technique which can function at room temperature, therefore the temperatures of feeding gases and the reactors were not controlled and close to ambient temperature (ca. 25 °C).

The DBD reactor was made of a quartz tube with an inner diameter of 40 mm, a wall thickness of 2.0 mm, and a length of 265 mm. The inner electrode, made of a stainless-steel rod with diameter of 3.4 mm, was aligned horizontally along the centerline of the reactor. The outer electrode was made of stainless-steel mesh and was wrapped around the outside of the crystal quartz tube. The length of the outer electrode, being equal to discharge length, was 170 mm. The discharge gap was 18.3 mm and the effective discharge volume was $212\,cm^3$.

The CPC reactor was constructed by packing catalysts into a DBD reactor. Each columnar catalyst was 5.4 mm long, 5.2 mm in diameter, and with a density of $1.25\,g/cm^3$. The commercial catalysts consisted of $CuO/ZnO/MgO/Al_2O_3$ with a mass ratio (%) of 64/24/2/10. The void fraction of the CPC reactor was ca. 0.48.

An alternating-current power supply with variable voltage and frequency was used as the power source. The voltage from power source was increased 150 times by a step-up transformer (Jui-Hsiang Pty Co. Ltd.). The final output voltage to generate plasmas was within 0–26 kV. The power consumption of the whole system was determined by integration of voltage and current (both in root-mean-square

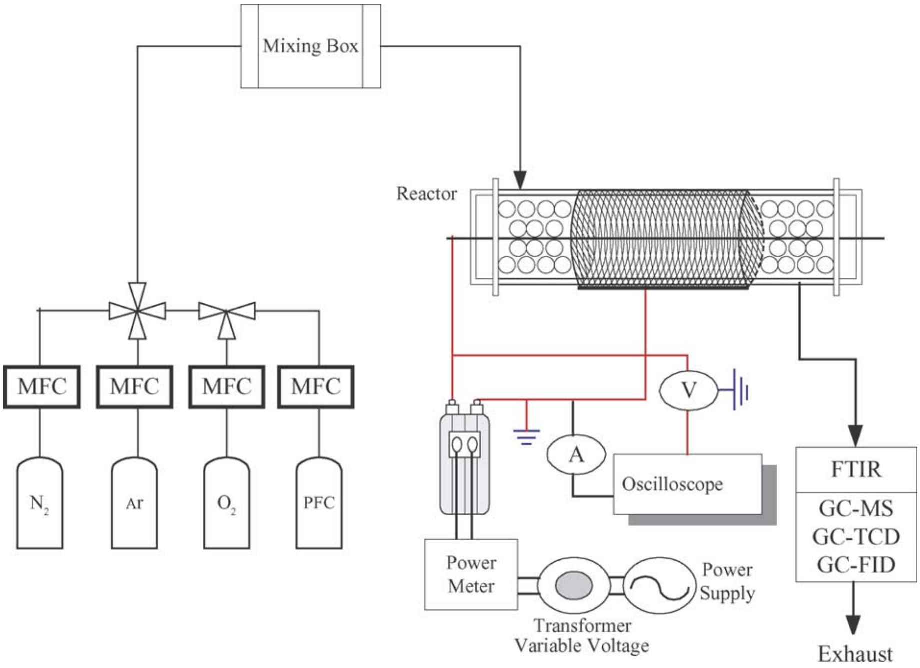

Fig. 2. Schematic of the experimental setup of the CPC for PFC abatement.

value) which were measured by a power meter installed in the power supply (Chen-Hwa, 2700P).

Identification and measurement of PFCs and associated end-products were carried out by an on-line Fourier transform infrared (FT-IR) spectrometer (Bio-Rad, FTS-165). The IR spectra spanning wavenumber ranging from 400 to 4000 cm^{-1} were recorded with a resolution of 2 cm. Each data point represents the average of 16 times' scans. A gas chromatograph (GC-TCD/FID, China Chromatography 9800) was used to check the accuracy of the IR analysis.

The removal efficiency (η), products' selectivity (S), and carbon balance value (B_c) are defined as follows:

- Removal efficiency:

$$\eta\,(\%) = \frac{[\mathrm{PFC}]_{\mathrm{in}} - [\mathrm{PFC}]_{\mathrm{out}}}{[\mathrm{PFC}]_{\mathrm{in}}} \times 100$$

  where $[\mathrm{PFC}]_{\mathrm{in}}$ is the concentration of PFCs in influent (ppmv), and $[\mathrm{PFC}]_{\mathrm{out}}$ the concentration of PFCs in effluent (ppmv).

- Selectivity:

$$S\,(\%) = \frac{C_i}{C_t} \times 100$$

  where C_i is the concentration of the carbon-containing product of interest in effluent (ppmv), and C_t the concentration of total carbon-containing products (excluding residual reactant) (ppmv).

- Carbon balance value

$$B_{\mathrm{c}} = \frac{\sum C_{\mathrm{out}}}{\sum C_{\mathrm{in}}}$$

where $\sum C_{\mathrm{out}}$ is the total carbons measured in outlet gas streams (mol), and $\sum C_{\mathrm{in}}$ the total carbons fed in reactor (mol).

3. Results and discussion

Three kinds of tests including catalysis only, plasmas only (DBD) and CPC are performed in this study. A CPC reactor is constructed with catalysts being packed into a DBD reactor. Experiment of catalysis is carried out with a CPC reactor without discharge. This study first focuses on CF_4 abatement because CF_4 is the most stable gas among the PFCs. One might reasonably anticipate that if CF_4 could be decomposed, all other PFCs would be effectively destroyed under the same conditions.

Fig. 3 shows the performances of CF_4 abatement by catalysis only, DBD only, and CPC, respectively. The gas flow rate was kept at 600 sccm. The feeding gases contained 300 ppm CF_4, 40% Ar, 20% O_2, and balanced N_2. All experimental runs were conducted under room temperature and atmospheric pressure. For the tests of DBD and CPC, a voltage of 15 kV with a frequency of 240 Hz was applied to generate plasmas. For the tests of catalysis only, the experiment was conducted with a CPC reactor without voltage being applied (without plasma). As shown in Fig. 3, performance of CF_4 abatement is in the order of CPC > DBD > catalysis. Catalysis is ineffective in CF_4 abatement (CF_4 removal efficiency is lower than 3%). The reason is mainly attributed to the low activity of catalysts being operated at room temperature. When a high voltage is supplied to the CPC reactor,

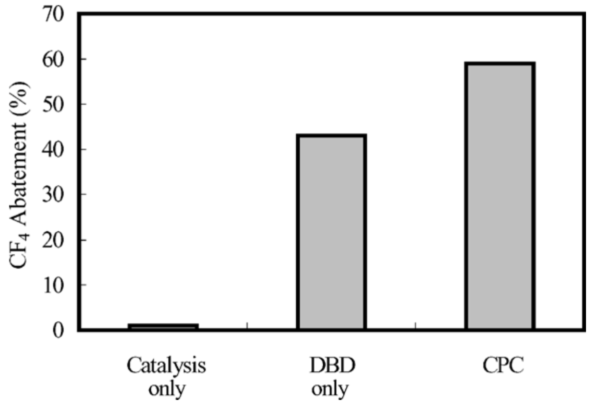

Fig. 3. Abatement of CF_4 at room temperature and atmospheric pressure by catalysis, DBD, and CPC, respectively. The inlet gas streams contain 300 ppm CF_4, 40% Ar, 20% O_2, and N_2 as balance. The gas flow rate is kept at 600 sccm. For CPC and DBD, applied voltage and frequency are 15 kV and 240 Hz, respectively.

the plasmas are induced, CF_4 removal efficiency increases instantly and 60% CF_4 is removed. On the other hand, CF_4 removal efficiency achieved by DBD is only 43%. In other words, CPC achieves 17% higher removal efficiency than DBD. These facts demonstrate that enhancement of CF_4 abatement is achieved by packing catalysts into a plasma environment.

The operation parameters of catalysis generally include gas temperature and pressure. In contrast, the adjustable parameters of nonthermal plasmas include applied voltage, frequency, and gas pressure. The aim of this study is to develop a technology that is functional at room temperature and atmospheric pressure. Therefore, the following experimental tests are conducted to evaluate the influences of applied voltage and frequency on DBD and CPC for CF_4 abatement. Fig. 4 shows the dependence of CF_4 removal efficiency on applied voltage. The feeding gas streams contained 300 ppm CF_4, 40% Ar, 20% O_2, and balanced N_2. The gas flow rate and applied frequency were kept at 600 sccm and 60 Hz, respectively. For the case of DBD, CF_4 cannot be removed until the applied voltage is higher than 7 kV because the plasmas are not yet induced. As applied voltage is increased from 9 to 21 kV, CF_4 removal efficiency increases linearly from 1 to 28%. Similar trend is observed for CPC experiment. However, the efficiency of CPC is about 5% higher than that of DBD. In brief, CF_4 removal efficiency increases with increasing applied voltage for both DBD and CPC. It is attributed to that a higher electric field is induced when a higher voltage is applied. Electrons accelerate in electric field and their kinetic energies increase in the meantime. These energetic electrons are suitable to decompose PFCs.

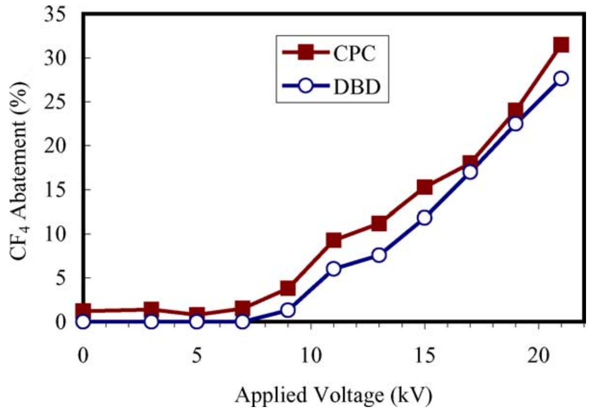

Fig. 4. CF_4 abatement by DBD and CPC as a function of applied voltage. The inlet gas streams contain 300 ppm CF_4, 40% Ar, 20% O_2, and N_2 as balance. The gas flow rate is kept at 600 sccm. The frequency of a.c. power is set to 60 Hz.

In addition to applied voltage, another important parameter for nonthermal plasmas is applied frequency. As applied frequency is increased, more power is deposited into the plasmas, resulting in a greater electron density and higher electron-impact reaction rates, which in turns of a higher destruction efficiency. In other words, a higher PFC removal efficiency can be achieved when a higher frequency is applied. Experimental results agree with this interpretation as shown in Fig. 5. The experimental conditions are kept the same as that shown in Fig. 4 except for applied voltage and frequency. The applied voltage was kept at 15 kV, while the frequency varied from 60 to 240 Hz. Difference of CF_4 removal efficiency between DBD and CPC is getting larger with the increase of applied frequency. For example, 5% difference is found between CPC and DBD operated at 60 Hz, and 15% at 240 Hz. This result implies that CPC being operated at a higher frequency favors CF_4 abatement.

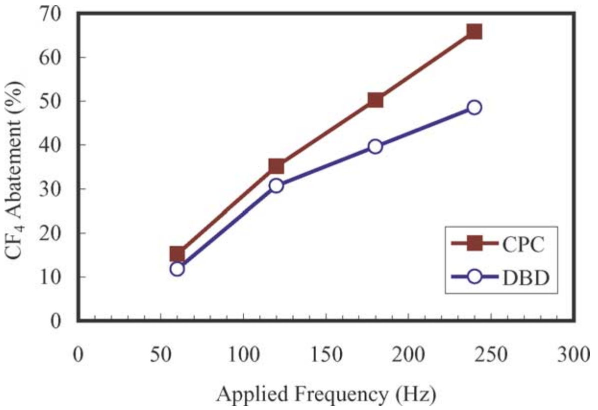

Fig. 5. CF_4 abatement by DBD and CPC as a function of applied frequency. The inlet gas streams contain 300 ppm CF_4, 40% Ar, 20% O_2, and N_2 as balance. The gas flow rate is kept at 600 sccm. The voltage of a.c. power is set to 15 kV.

Fig. 6 shows the dependence of CF_4 removal efficiency on oxygen content varying from 0 to 20% by volume. The inlet gas streams contain 300 ppm CF_4, 40% Ar, select O_2 content, and balanced N_2. The gas flow rate is kept at 600 sccm. For CPC and DBD, applied voltage and frequency are kept

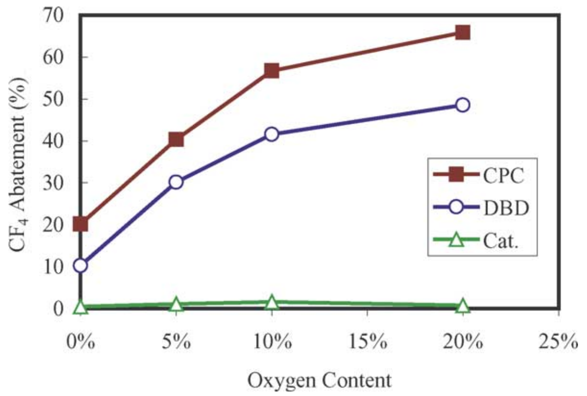

Fig. 6. Influence of oxygen content on CF_4 abatement by catalysis (Cat.), DBD, and CPC. The inlet gas streams contain 300 ppm CF_4, 40% Ar, select O_2 content, and balanced N_2. The gas flow rate is kept at 600 sccm. For CPC and DBD, applied voltage and frequency are kept at 15 kV and 240 Hz, respectively.

at 15 kV and 240 Hz, respectively. For the case of CF_4 abatement by catalysis only, removal efficiency is relatively low regardless oxygen content. On the other hand, removal efficiency increases with increasing oxygen content for both DBD and CPC. In plasmas, O atoms are generated via electron-impact reactions, $O_2 + e \rightarrow O + O + e$. Hence, the more oxygen content in the feeding gas, the more O atoms will be formed. Chemically active O atoms can react with CF_3 radicals to inhibit the recombination to form CF_4 or C_2F_6 (Reactions (5)–(7)). Fig. 6 also demonstrates that CF_4 abatement by CPC is greater than that by DBD. This result indicates that the packed catalysts do not suffer adverse effect with the presence of oxygen.

Previous results demonstrate that CF_4 abatement by CPC can be functional under room temperature. In addition, CPC has better performances than DBD. The latter part of the study is going to evaluate the performance of CPC for other PFCs (see Fig. 7). The operation conditions are controlled the same as previous tests. Concentration of each PFC is fixed at 300 ppm. The destruction efficiency of four kinds of PFCs is in the order of $NF_3 > SF_6 > C_2F_6 > CF_4$. This sequence is in the reverse order of bond energy of $N–F < S–F < C–F$. A weaker bond is easier to decompose. The maximum removal efficiency achieves 66% for CF_4 at a frequency of 240 Hz; while 83% for C_2F_6. Complete conversions are achieved for SF_6 and NF_3 at a frequency of 150 Hz.

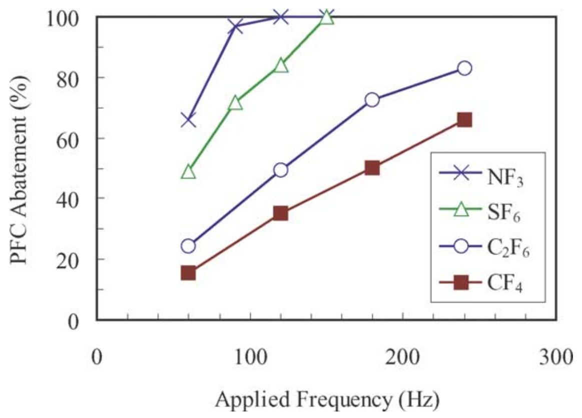

Fig. 7. Abatement of CF_4, C_2F_6, SF_6 and NF_3 with CPC. The inlet gas streams contain 300 ppm PFCs, 40% Ar, 20% O_2, and balanced N_2. The gas flow rate is kept at 600 sccm. The applied voltage is kept at 15 kV.

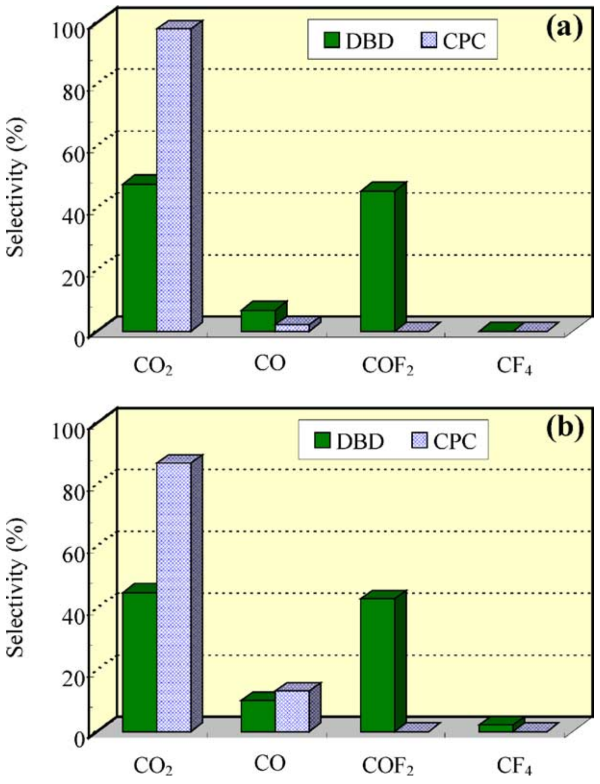

Fig. 8. Products' selectivity for CF_4 (a) and C_2F_6 (b) abatements by DBD and CPC. The inlet gas streams contain 300 ppm CF_4 or C_2F_6, 40% Ar, 20% O_2, and balanced N_2. The gas flow rate is kept at 600 sccm. The applied voltage and frequency are kept at 21 kV and 60 Hz, respectively.

The selectivity of products formed in destroying CF_4 with DBD and CPC is shown in Fig. 8(a). The carbon balance value was 0.98. The gas streams contained 300 ppm CF_4, 40% Ar, 20% O_2, and balanced N_2. The major products formed in DBD process include CO_2, COF_2 and CO. The carbons of CF_4 are converted to CO_2 and COF_2, corresponding to 48 and 45%, respectively. Formation of COF_2 is speculated through gas-phase radical reactions, e.g. $CF_3 + O \rightarrow COF_2 + F$; $CF_2 + O_2 \rightarrow COF_2 + O$. Carbon monoxide is mainly formed by electron impact with CO_2 and/or COF_2, $e + CO_2 \rightarrow CO + O + e$; $e + COF_2 \rightarrow CO + F_2 + e$. Fig. 8(a) also shows the selectivity of products in the CPC experiment. The carbon balance value was 0.93. Major products include CO_2 and a little CO. Interestingly, COF_2 which had a high selectivity in DBD experiments was not found in CPC experiments.

Fig. 8(b) shows the selectivity of products achieved with DBD and CPC for C_2F_6 abatement. The major products identified by FT-IR include CO_2, COF_2, CO, and CF_4. These chemicals, except for CF_4, formed in C_2F_6 processing are the same as the products formed in CF_4 processing. CF_4 are generated from recombination of CF_3 and F radicals. The major products formed in DBD process include CO_2, COF_2, CO and CF_4, corresponding to 45, 43, 10, and 2%, respectively. Fig. 8(b) also shows the selectivity of products in the CPC experiment. Again, neither COF_2 nor CF_4 can be found in CPC experiment.

In brief, the major products formed in DBD process are CO_2, COF_2 and a little CO. A little CF_4 is also formed in C_2F_6 abatement. COF_2 is a toxic gas (TLV $= 2$ ppm), and CF_4 is a greenhouse gas. These unwanted chemicals must be further treated. In contrast, the major products in CPC process are CO_2 and a little CO, which are less harmful to human health and the environment.

4. Conclusions

This study investigates the enhancement of PFC abatement by combining catalysis with plasma technologies. CPC is functional to PFC abatement at room temperature and atmospheric pressure. PFC destruction efficiency achieved by CPC is greater than that achieved by either catalysis only or plasma only. In addition, products in CPC process are less harmful to human health and the environment compared with the products formed in DBD process. Experimental results demonstrate that CPC is a good means for PFC abatement. Complete destructions are achieved for processing SF_6 and NF_3 by CPC. Removal efficiencies for CF_4 and C_2F_6 are 66 and 83%, respectively. Although the efficiencies for CF_4 and C_2F_6 abatements are not high enough, it must be pointed out that the current CPC system has not been optimized. Studies on CPC operation at elevated temperature and reactor modification to enhance PFC removal efficiency are on going.

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