Chapter 5
Plasma-Catalytic Removal of NO_x in Mobile and Stationary Sources

Ahmed Khacef and Patrick Da Costa

5.1 Introduction

Nitrogen oxides, which refer specifically to NO_x (NO and NO_2), are the major pollutants in the atmosphere from industries (mobile or stationary sources), which lead to acid rain and photochemical smog, and have been shown to be detrimental to human health and the environment. Automobiles and other mobile sources contribute to about 50% of the NO_x that is emitted in the atmosphere, and nitrogen monoxide (NO) is the major portion of it [1, 2].

Lean-burn engines are receiving increasing application because of their promise of improved fuel economy over stoichiometric conditions. However, they offer special challenges for meeting increasingly stringent emissions standards because of the difficulty of removing NO_x in an oxidizing environment where traditional three-way catalysts cannot work.

Consequently, several methods collectively called deNO_x processes have been developed to treat the effluents of mobile and power plants sources. Among them, selective catalytic reduction (SCR) and selective non-catalytic reduction (SNCR) are currently used to convert NO_x into nitrogen (N_2) molecules [3, 4]. Nowadays, the selective catalytic reduction of NO_x by urea (urea-SCR) and lean-NO_x trap technology (LNT) are the two most efficient NO_x reduction technologies currently available for diesel engine emission control [5–8]. Both technologies provide high NO_x reduction performance over a wide temperature range (200–400 °C) under well-controlled conditions but still have serious drawbacks [9–19].

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As an alternative to catalytic processes which require high temperatures nonthermal plasma (NTP) also referred as "non-equilibrium plasma" or "cold plasma," such dielectric barrier discharges (DBDs) and corona discharges (CD) have been extensively investigated in the field of pollution control and frequently proposed in the literature for the removal of volatile organic compounds (VOCs), NO_x, and SO_2 [20–26].

In NTP, background gaseous species are chemically excited or dissociated directly by electronic impact, while the temperature of the reactants (i.e., gas temperature) remains relatively low, and thus the product distributions far from the chemical equilibrium may be obtained. In that case, the most useful deposition of energy is associated with the production of excited species (atoms, molecules) and activated species (radicals, ions) that eventually lead to the chemical conversion of pollutants. Although NTPs present attractive properties (low temperature, atmospheric pressure, compactness, low cost) and a unique way to induce gas-phase reactions by electron collisions, the formation of unwanted by-products and poor energy efficiency are serious obstacles toward their industrial implementation. To overcome these drawbacks, a more effective use of NTP is possible by exploiting its inherent synergetic potential through combination with heterogeneous catalysts as emphasized by different groups [27–29]. This innovative technique called plasma catalysis that combines the advantages of both NTP and catalysis has become a hot topic over the last decade. Catalyst can be combined with plasma in two ways: in-plasma catalysis (IPC), with the catalyst directly into the discharge zone, or post-plasma catalysis (PPC), with the catalyst downstream the discharge zone.

The conventional NTP reactors that are widely used for various environmental applications are subdivided according to the type of discharge mode (pulse, DC, AC, RF, microwave), presence of a dielectric barrier or catalyst, and geometry (cylinder, plane). It is important to note that the chemical potential of each discharge mode differs enormously from one discharge to another. Roughly speaking, the efficiency of a plasma discharge to remove pollutant from gas stream depends mainly on its ability to produce large amount of active species in the plasma.

In an attempt to overcome the inherent shortcomings of these existing technologies, plasma- assisted catalysis for lean-NO_x reduction through the improvement of the hydrocarbons (HC)- SCR technology has emerged as a promising alternative to the more mature urea-SCR and LNT technologies [30–37].

5.2 State of the Art on Catalytic NO_x Abatement Systems

Since 1970, NO_x abatement technologies were majorly based on selective catalytic reduction with ammonia (NH_3-SCR). Nowadays, two mature technologies are used at least for mobile sources: NO_x trap or NO_x storage/reduction (NSR) and NH_3-SCR [5–8]. Other promising technologies such as HC/HCR are still studied in order to use the reducing agent present in the feed for reducing the NO_x in situ.

The NO_x trap is one of the actual technologies already in used in automotive posttreatment lines. The used materials are based on noble metal dispersed on a support such as alumina or ceria zirconia containing an adsorbent material such as barium oxide. Noble metal can be deactivated or poisoned since sulfur is present in the feed. The deactivation mechanisms of NSR catalysts, especially the sulfur poisoning and thermal degradation, were already extensively reviewed. Finally, recent developments of NSR catalysts were addressed in detail, concentrating on the improvements over precious metals, NO_x storage materials, and metal oxide supports.

Despite the first-generation NSR catalysts being quite successful in NO_x emission control, new-generation NSR catalysts with low cost, high efficiency, and durability are urgently needed to meet the ever rigorous NO_x emission regulations and develop the NSR technology. Thus, the development of new materials (e.g., perovskite oxide-based materials) [38] as alternatives of precious metals is very promising to reduce the cost of NSR catalysts. Finally, a novel synthesis method and a novel structured NSR catalyst (e.g., 3-D structured catalysts) would help to increase the NSR activity, sulfur poisoning resistance, and thermal stability needed for the NSR regeneration [39].

The SCR of NO_x with NH_3 was also considered for a long time to be the most efficient technology for reducing NO_x emission in the presence of excess oxygen [40]. The V_2O_5-$WO_3(MoO_3)/TiO_2$ material has been commercially employed as a NH_3-SCR catalyst for a number of years. However, several serious problems with this catalyst still remain, e.g., the narrow temperature window (i.e., only applicable in 300–400 °C), the high activity of SO_2 oxidation, and the toxicity of V_2O_5 [41]. Therefore, new catalysts with environmentally benign characteristics and high SCR performance in a wide temperature range are required. Since the discovery of Cu-ZSM-5 catalyst as an efficient catalyst for NO_x removal in 1986 by Iwamoto et al. [42], copper- or iron-exchanged zeolites have been and are still widely investigated [43]. However, the major disadvantage of zeolite-based materials is their poor resistance to high temperature treatment. New zeolitic systems such as SSZ-13 and SAPO-34 are in development to give to such system high SCR performance even after hydrothermal aging [44–47].

For SCR of NO_x with HC, according to Burch et al. [5], more than 1000 catalysts were tested in laboratory, and more than 80% NO_x conversion was obtained over specific temperature ranges under lean-burn conditions. It was never possible to translate this laboratory success to real exhaust automotive systems. The authors proposed three factors in order to explain that point:

(i) Laboratory tests mostly focused on pure reducing agents such as the lower alkanes and alkenes, whereas onboard reducing agents, such as gasoline or diesel, contain hundreds of compounds, some of which may poison or inhibit the SCR reaction.
(ii) The space velocities used in laboratory micro-reactors are frequently much lower than those found in real exhausts.
(iii) The temperature range in an engine tests is much wider than the narrow window of high activity observed in typical laboratory tests.

Thus, coupled plasma catalysis technologies could be a solution to the points (i) and (iii).

5.3 Nonthermal Plasma Reactors for NO_x Treatment

Nonthermal plasmas for chemical treatment of industrial exhaust gases may be produced by a variety of electrical discharge reactors (corona discharge, surface discharge, dielectric-barrier discharge, etc.) [48–51] or by electron beam irradiation [52]. The electron beam technique, which has been first used, is based on irradiation of the gas flow by high energetic electrons being generated in ultrahigh vacuum accelerators with voltages ranging from tens of kV to some MV and transmitted into the gas flow through titanium foils. Each of the primary beam electrons generates a large number of secondary electrons in ionizing collisions, which have the right energy for efficient radical generation finally resulting in efficient pollutant removal. For some industrial and domestic applications, the pulsed corona and DBD reactors are much more suited than e-beam devices because of their high selectivity, moderate operating conditions (atmospheric pressure and room temperature), and relatively low maintenance requirements resulting in relatively low energy costs of the pollutant treatment.

5.3.1 Corona Reactors

Corona discharge is a transient luminous discharge that appears in regions of high electric field near sharp points, edges, or wires in electrically stressed gases prior to the point of electrical breakdown. The corona discharges are always nonuniform: a strong electric field, ionization, and luminosity are located in the vicinity of one electrode. The corona can be observed, for example, around high-voltage transmission lines, around lightning rods, along wire surrounded by a coaxial cylinder, or near irregularities in the form of sharp points, on the surface of a conductor at high voltage. The main physical and engineering principles of the corona discharges can be found in Loeb [53], Fridman et al. [54], and Rutgers and van Veldhuizen [55].

Various reactor configurations are used to generate corona discharges. In laboratory studies of corona discharges, the most common geometry is a pin-plane geometry (Fig. 5.1), where a needle is placed above a grounded plane. The high voltage is applied to the needle electrode. However, for industrial applications, this geometry is not sufficient, as it does not fill the whole gas volume with the discharge. The most popular geometries are the multi-pins-plane, wire- cylinder, and the saw-blade geometries [56, 57] as in electrostatic precipitators. The wire- cylinder geometry is probably used the most. It ensures a quite homogeneous distribution of the discharge that maximizes the active discharge volume and is easy to implement

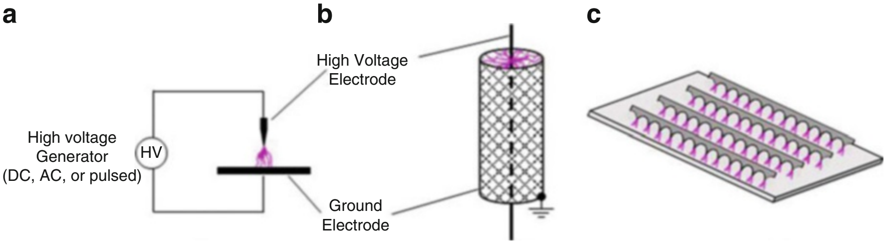

Fig. 5.1 Common corona reactor geometries: (**a**) pin-plane, (**b**) wire-cylinder, and (**c**) saw-blade

in a gas flow system. Often, multiple wire-cylinder reactors are mounted in parallel with regard to the gas flow to enable high gas throughput.

Corona discharges are separated into two different categories: continuous and pulsed. Continuous corona discharges occur at DC or low-frequency AC voltages. A recent example of work on DC-excited corona discharges is given by Eichwald et al. [58]. Application of the continuous coronas is limited by low current and power, which results in a low treatment rate of exhaust gas. Increasing the corona power could result in a large current short circuit, and a large amount of NO_x can be produced when spark breakdown occurs. For the purpose of gas cleaning, spark breakdown should be avoided.

Increasing the corona power without transition to sparks becomes possible by using voltage pulses having nanosecond or sub-microsecond duration. Since discharge gaps of several tens centimeters can be used, the pulsed corona discharge (PCD) reactor is ideal for treatment of very large gas flow rates [59–61]. The costs of the PCD reactor are low compared to a DBD reactor for the same gas flow rate. However, the development of cost-effective pulsed power supplies is challenging. Further, for good efficiency and long lifetime of the power supply, care has to be taken for good electrical matching between power supply and reactor [62].

PCD reactors have been considered for the removal of pollutants both in the gas phase [48, 60, 63] and in the liquid phase [64]. A combination of pulsed corona with catalysts can be practical for applications to achieve improved results in the treatment of automotive exhausts and for hydrogen production from heavy hydrocarbons. Another interesting technological hybrid is related to the pulsed corona coupled with water flow. Such a system can be arranged either in the form of a shower, which is called a spray corona, or with a thin water film on the walls, which is usually referred to as a wet corona.

Figure 5.2 shows an example of pulsed corona discharges developed from each pin in pure N_2 at atmospheric pressure for a multi-pins-plane corona reactor. The illuminating paths indicate the position where the streamer heads have moved. Below a certain applied voltage, the corona discharge filaments are almost invisible to the human eyes, and only at the optimum voltage, which is not sufficient to generate arc discharge, and after a long adaptation in the dark, they can just be observed. The practical advantages of the PCD are that the short duration of the pulse ensures that no transition to spark takes place; therefore, it can be used at voltages

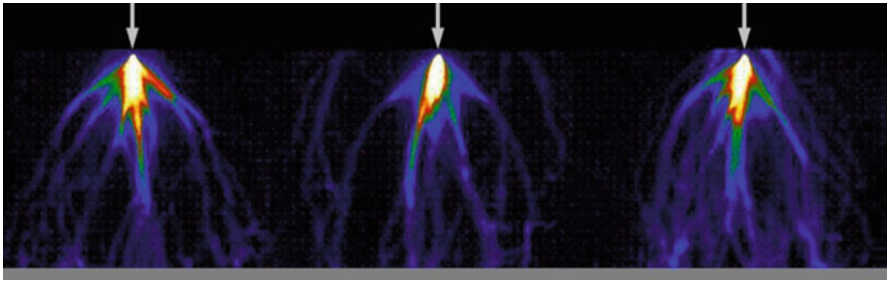

Fig. 5.2 Streamer discharges in 12 mm gap in N_2 at atmospheric pressure with a 16 kV pulse, 30 ns rise time, and half-width of about 80 ns (multi-pins-plane corona reactor) [63]

and currents higher than that at continuous corona can be used. Large voltages lead to higher electric fields and electron temperatures, which increases ionization and dissociation rates. Additionally, due to the short pulse duration, only electrons are significantly accelerated in few or tens of nanoseconds, and the gas heating can be kept to a minimum.

5.3.2 Dielectric Barrier Discharge Reactors

Another approach that avoids spark formation in streamer channels and instabilities in space (both effects are undesired in a volume discharge for chemical conversions) is based on the use of at least one electrically insulating barrier between the electrodes. The introduction of a dielectric barrier between the electrodes limits the direct current. Such discharges are referred to as dielectric barrier discharges or silent discharges (SD). The electrical circuit of the discharge reactor can be represented by the dielectric barrier capacity being in series with the discharge gap represented by the variable ohmic impedance of the gas discharge plasma paralleled by the capacitance of the discharge. Because of the capacitive coupling of the current to the discharge gap, DBD reactors have to be operated with AC or pulse repetitive voltages. Further reliable operation requires high-quality, low dielectric loss barrier materials having a high dielectric breakdown strength (>10 kV mm^{-1}) and high bulk resistivity ($>108\ \Omega$ cm^{-1}). In case of low temperatures, polymeric barrier materials such as PTFE can be used. For exhaust gases having temperatures higher than 100 °C, rather expensive densely sintered ceramics or quartz glass barriers are required. An advantage of DBD reactors is that barriers having certain catalytic properties can be used for plasma-catalytic hybrid treatment of gases.

DBD processing is a very mature technology, first investigated by Siemens [65] in the 1850’s for ozone generation. Ozone formation requires three criteria: (i) energetic electrons that are able to efficiently dissociate O_2 (4–5 eV, see Fig. 5.4), (ii) high pressure because of the three-body reaction which is responsible for ozone production, and (iii) low gas temperature because of the reduced lifetime of ozone at high gas temperature. DBDs can deliver all these requirements as the bulk gas temperature can be maintained close to room temperature (for high power, cooling the electrodes is required), while the electron temperature is typically 2–5 eV. DBDs are now routinely used in different industrial and fundamental applications such as water purification, polymer treatment, UV light generation, biological and medical treatment, pollution control, and exhaust cleaning from CO, NO_x, SO_x, and VOCs. DBDs could be generated in parallel-plane or in coaxial cylindrical reactor geometries, very common in ozonizers and other cases of gas treatment, as illustrated in Fig. 5.3. The flexibility of DBD configurations with respect to geometrical shape, operating medium, and operating parameters (input power, frequency, and gas flow) is remarkable. In many situations, discharge conditions optimized in small laboratory experiments can be scaled up to large industrial installations.

Kogelschatz, Eliasson, and their group at ABB [67–69] have greatly contributed to the fundamental understanding and industrial applications of DBDs. Typical characteristics of the DBD micro-discharges in a 1 mm gap in atmospheric air are summarized in Table 5.1.

Atmospheric pressure DBDs usually are filamentary. However, in certain gas mixtures, a quasi- homogeneous glow mode can be observed [70, 71]. Since the plasma-chemical efficiency of DBD reactors decreases with increasing discharge gap, typical discharge gap widths are in the range of a few millimeters only. When pulsed excitation with sub-microsecond rise time is applied, the discharge gap of DBD reactors can be increased from typically 1 mm to about 1 cm without loss in plasma-chemical efficiency [72]. Nevertheless, the treatment of industrial-scale gas flows requires a lot of paralleled discharge gaps in order to keep the flow resistance low.

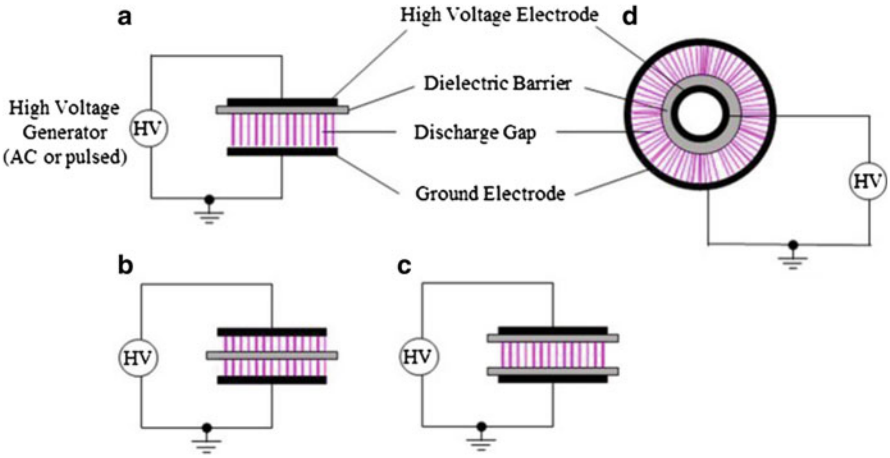

Fig. 5.3 Common dielectric barrier discharge electrode configurations: (**a**–**c**) planar and (**d**) cylindrical

Table 5.1 Characteristic of DBD in air at atmospheric pressure

Duration	10^{-9}–10^{-8} s
Filament radius	About 10^{-4} m
Peak current	0.1 A
Current density	10^{6}–10^{7} A m^{-2}
Total charge	10^{-10}–10^{-9} C
Electron density	10^{20}–10^{21} m^{-3}
Mean electron energy	1–10 eV
Filament temperature	Close to average gas temperature in the gap

5.4 NO_x Chemistry in Nonthermal Plasma

The energetic electrons produced in the plasma discharge mainly initiate the plasma chemistry. The elementary process of radical formation and reactions in NTPs can be broadly divided into a primary process and a secondary process based on the timescale of streamer propagation [73]. The primary process that has a typical timescale in the range of tenths of nanoseconds includes ionization, excitation, dissociation, light emission, and charge transfer. The efficiency of the primary process is highly dependent on energization methods and their parameters, such as nature of the power source (pulse, DC, or AC), voltage rise time, duration, and frequency. The secondary process is the subsequent chemical reactions involving the products of primary processes (electrons, radicals, ions, and excited molecules) to form additional radical species and reactive molecules (O_3, HO_2, and H_2O_2) by radical-neutral recombination. The secondary process is usually completed within approximately milliseconds. Oxidation is dominant for plasma processing of exhausts (with or without hydrocarbons) containing dilute concentrations of NO_x (mainly NO with concentrations ranged from few ppm to hundreds ppm) in N_2-O_2-H_2O mixtures, particularly when O_2 concentration is 5% or higher.

5.4.1 Plasma Without Hydrocarbons

In NTP treatment of NO-N_2-O_2-H_2O mixtures, the kinetic energy of the electrons is mainly deposited into the major gas components N_2, O_2, and H_2O. The result is the generation of N and O radicals through electron-impact dissociation:

$$e + O_2 \rightarrow e + O\left(^3P\right) + O\left(^3P, {}^1D\right)$$

$$e + N_2 \rightarrow e + N\left(^4S\right) + N\left(^4S, {}^2D\right)$$

where O(^{3}P) and O(^{1}D) are the ground-state and metastable excited-state atomic oxygen radicals, respectively, and N(^{4}S) and N(^{2}D) are ground-state and metastable excited-state atomic nitrogen radicals, respectively. The $N(^4S)$ is the only plasma-produced species that could effectively lead to the chemical reduction of NO [66, 74] accomplished via:

$$N\left(^4S\right) + NO \rightarrow N_2 + O$$

The O radical resulting from previous reaction can lead to the oxidation of NO to NO_2. However, the amount of O radicals is, as most, equal to the number of $N(^4S)$ produced in the plasma. Under this condition, most of the NO react with $N(^4S)$, and only a very small amount of NO_2 is produced [75].

Most experimental investigations focused on the oxidation of NO to NO_2 with different gaseous mixtures and energization systems. It was found that the oxidation of NO to NO_2 significantly depends on gaseous mixtures. It is very important to make a distinction between NO removal by chemical oxidation and NO removal by chemical reduction. Oxygen amount in the gas mixtures could greatly suppress the reduction process and enhance the oxidation process or the opposite. Yan et al. [76] show that the reduction process-induced NO removal can be negligible in the presence of O_2 with concentration of 3.6% of higher, which means that NO is completely converted into NO_2. In that case, the oxidation pathway becomes dominant for two reasons:

(a) The dissociation energy of O_2 (4.8 eV) is lower than that of N_2 (9.2 eV). For common atmospheric pressure electrical discharge plasma, the average electron kinetic energy is relatively low (3–6 eV) [77–79]. Under this condition, the rate for electron-impact dissociation of O_2 is much higher compared to that of N_2 as shown in Fig. 5.4.

(b) High electron energies are required to optimize the production of $N(^4S)$ atoms by electron-impact dissociation of N_2. Under conditions optimum for the dissociation of N_2, a large number of excited nitrogen atoms, $N(^2D)$, is produced [80]. The $N(^2D)$ species can lead to undesired reactions in the presence of O_2. Rather than reducing NO to N_2, the $N(^2D)$ species would react with O_2 to produce NO.

If oxygen concentration is less than 1%, the reduction process is dominant as showed in pulsed corona discharge under high specific input energy (SIE > 30 Wh/m) [81]. In that case, the amount of NO_2 produced could be larger than the removed NO because of $N(^2D)$-induced NO formation and NO to NO_2 conversion. The back conversion of NO_2 to NO becomes a limiting factor for the NO oxidation efficiency and energy cost. As reported by Tas et al. [82], the energy required for this process ($\geq$ 100 eV/molecule) is one order of magnitude higher than that for the oxidation process (50 eV/molecule) in the gas-phase reaction.

Additionally, the secondary radical species such as HO_2, NO_3, and O_3 are produced and consumed on longer timescales than the primary radicals. Ozone generated at low temperature through three-body reaction of $O(^3P)$ ($O(^1D)$ in humid gas) with O_2 is quickly quenched by H_2O to form hydroxyl radicals OH

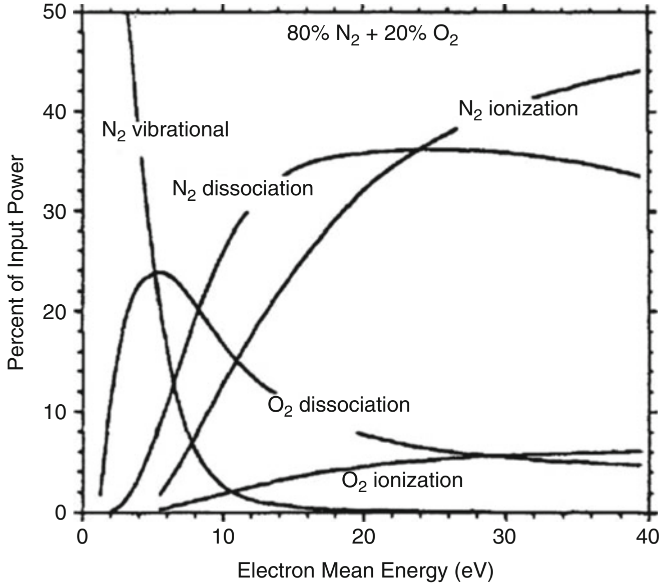

Fig. 5.4 Electrical power dissipation in a dry air discharge, showing the percent of input power consumed in the electron-impact processes leading to vibrational excitation, dissociation, and ionization of N_2 and O_2 [66]

which play an important role in the chemistry of NTP. As the oxygen and water vapor concentrations were increased, the rate of generation of OH and HO_2 radicals by reactions of atomic oxygen with H_2O molecules increases [22, 83]. These radicals (which density is highly sensitive to plasma conditions) initiate the reactions leading to chemical conversion of pollutant molecules (e.g., NO to NO_2 and NO_2 to nitric acid (HNO_3)). Figure 5.5 shows the production of oxidizing agents (O, OH, and O_3) was predicted by a self-consistent 0D-model in N_2-O_2-H_2O mixture at room temperature and atmospheric pressure [84]. O and OH maximum densities are achieved in some tens of nanoseconds, whereas the density of ozone slowly increases to reach a maximum about 1 ms after the plasma ignition.

In an oxygen-free mixture, the chemical kinetic is easier to study, but the situation is not representative of the engine or power plant exhaust gas emissions. The kinetic model in photo- triggered discharges of Fresnet et al. [85] predicts that N_2(a') singlet metastable states (which are strongly mixed by collisions and highly populated) represent an important pathway for NO removal in N_2/NO mixtures, besides NO reduction by N atoms. The drastic decrease in NO removal observed experimentally in the presence of water was attributed to de-excitation of the N_2(a') states by the H_2O molecules. For pulsed corona discharge in similar mixture, the kinetic model of Zhao et al. [86] considered reactions of NO and NO_2 with N and O atoms and with excited $N_2(A^3\Sigma)$ molecules and found that $N_2(A^3\Sigma)$ molecules are important as well, especially for the conversion of N_2O to N_2.

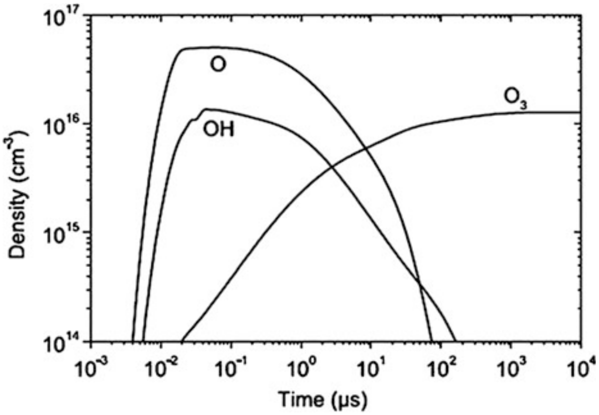

Fig. 5.5 O, OH, and O_3 densities as a function of time predicted by a 0D self-consistent model of photo-triggered discharge: N_2-O_2-H_2O (77.6-20-2.4) at 25 °C, 1 bar, and SIE = 100 J/L [84]

5.4.2 *Plasma with Hydrocarbons*

It is well known that, related to NTP deNO_x, promotion in the NO oxidation can be achieved with the addition of hydrocarbons. Hydrocarbon addition leads to substantial saving in the energy cost for deNO_x treatment. Among the possible hydrocarbons, ethylene (C_2H_4) [87–89], propene (C_3H_6) [1, 22, 87, 89, 90], and propane (C_3H_8) [87, 90] were selectively investigated to estimate their usefulness for the NO-NO_2 conversion. With the addition of hydrocarbons, the atomic oxygen produced in the plasma is considered to be the main initiator of the hydrocarbon chemistry, and the reactions typically produce intermediates such as hydroxy (OH) and peroxy radicals RO_2 (with R = H or C_xH_y).

The chemistry of NTP with hydrocarbons is complex and not fully understood. In the literature, there are some recommendations according to the channels and the products of the reaction. As it was remarked in the experimental work of Tsang [91], the total rate constant of the C_3H_6 + O reaction is quite accurate, and the most serious uncertainties are in the branching ratios. Usually, authors use different reaction channels and branching ratios in the modeling. Penetrante et al. [1], Shin and Yoon [87], and Park et al. [92] included the data of Tsang [91] and Wilk et al. [93] in the oxidation chemical kinetic. The effect of C_3H_6 on the conversion pathways of NO_x in pulsed corona discharge reactor was examined, and results reported by Penetrante et al. [1] show that in the very early stages of reaction, the propene is mainly consumed by O atoms following the three reaction channels:

$$\begin{aligned} C_3H_6 + O &\rightarrow C_2H_5 + HCO \\ &\rightarrow CH_2CO + CH_3 + H \\ &\rightarrow CH_3CHCO + H + H \end{aligned}$$

However, after the initial stages of the reaction chain, and following the production of hydroxyl radicals in the above reaction, the OH radical rather than the O atom becomes the main propene consuming species. The reaction products from these steps form hydrocarbon intermediates (CHO, CH_3O, CH_2OH, etc.) that react with oxygen to form HO_2 radicals. Chemical kinetic analysis shows that HO_2 radicals are the main species responsible for conversion of NO to NO_2. The main end products predicted by the mechanism of Martin et al. [94] are CO, CO_2, formaldehyde (CH_2O), and acetaldehyde (CH_3CHO), with smaller concentrations of ketene (CH_2CO) and methyl ketene (CH_3CHCO).

Dorai and Kushner [90, 95, 96] used a recommendation of Atkinson [97] to develop a mechanism in humid exhaust gas. The reaction of O with C_3H_6 they used differs from those suggested by Penetrante et al. [1] by the inclusion of the steps:

$$\begin{aligned} C_3H_6 + O &\rightarrow C_3H_6O \\ &\rightarrow CH_3CH_2CHO \\ &\rightarrow CH_2CHO + CH_3 \\ &\rightarrow C_2H_5 + HCO \end{aligned}$$

For the reaction of OH with propene, only the following step is considered:

$$C_3H_6 + OH \rightarrow C_3H_6OH$$

End products such as formaldehyde (CH_2O), acetaldehyde (CH_3CHO), methyloxirane (C_3H_6O), and propanal (CH_3CH_2CHO) were predicted as major by-products from propene-induced NO to NO_2 conversion. Experimental analysis of exhaust within similar composition made by Hoard and Panov [98] showed no presence of methyloxirane and propanal.

In the work of Filimonova et al. [89], the choice of reaction channels and their ratios was based on the experimental investigation of branching ratios in the C_3H_6 + O reaction obtained by Koda et al. [99]. Taking into account the measured yields of products and a pressure dependence of CH_2CHO formation, they used the following distribution:

$$\begin{aligned} C_3H_6 + O &\rightarrow CH_3 + CH_2CHO \\ &\rightarrow C_2H_5 + CHO \\ &\rightarrow C_2H_4 + CH_2O \end{aligned}$$

Regarding these three significant examples, an ambiguity about the choice of critical reaction channel (C_3H_6 + O) for NO_x oxidation needs to be clarified by additional experimental investigations and further refinement of the rate constants. In most cases, the comparison between experimental and simulation results is not suitable because the data is obtained in different conditions. However, some of the end products predicted by simulation results such as acetaldehyde, formaldehyde, methanol, methyl nitrate and nitrite, nitromethane, propylene oxide, and formic and nitric acids agree with experimental measurements as shown in the Figs. 5.6 and 5.7.

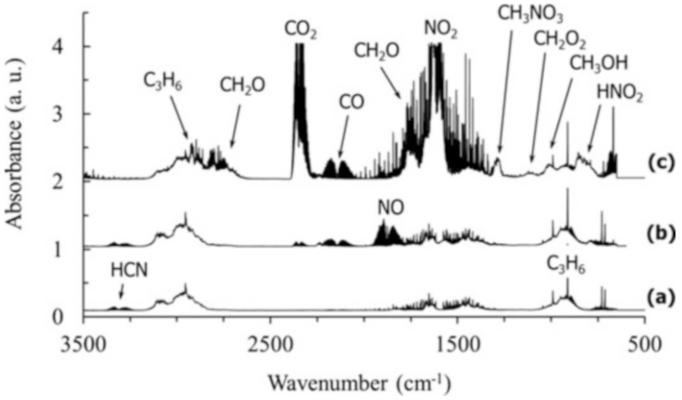

Fig. 5.6 Typical FTIR spectra produced by plasma processing (27 J/L) at room temperature of (a) C_3H_6 (500 ppm)-N_2, (b) NO (500 ppm)-C_3H_6 (500 ppm)-N_2, and (c) O_2 (10%)-NO (500 ppm)-C_3H_6 (500 ppm)-N_2 [100]

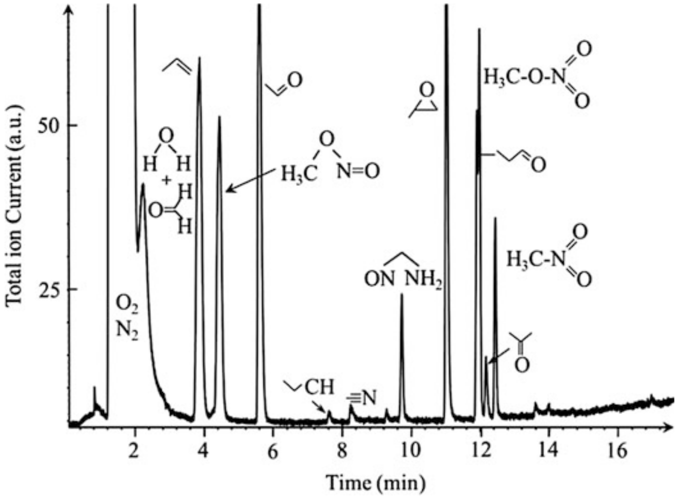

Fig. 5.7 Typical chromatogram plot of a gas phase from pulsed DBD processing O_2 (8%)-NO (300 ppm)-C_3H_6 (150 ppm)-N_2 (balance): SIE = 36 J/L, and $T = 150$ °C [101]

From these studies, the reaction products from these steps form hydrocarbon intermediates that react with oxygen to form HO_2 radicals that become the main component in the NO removal in the presence of C_3H_6 and C_2H_4. However, C_3H_6 is more beneficial compared with C_2H_4 for the cleaning process because of an additional formation of HC radicals.

For a given specific input energy, the gas temperature affects the efficiency of oxidation of NO to NO_2. Penetrante et al. [1] show that in gas mixtures without hydrocarbons, the efficiency of oxidation of NO to NO_2 by O radicals drops as the

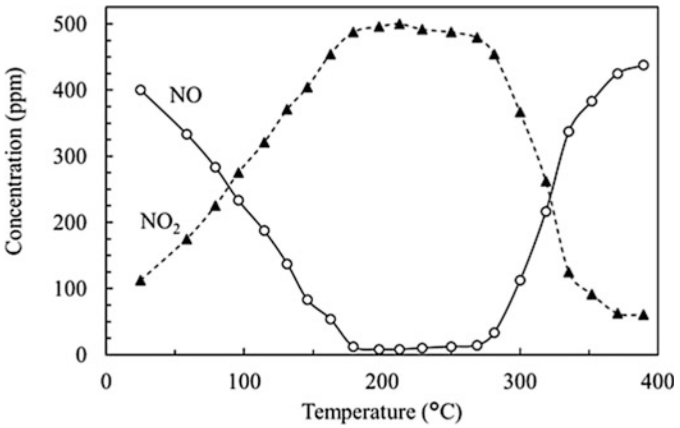

Fig. 5.8 NO and NO_2 concentrations as a function of gas temperature. Plasma processing of C_3H_6 (660 ppm)-NO (500 ppm)-O_2 (8% vol.)-N_2 at SIE = 36 J/L [101]

gas temperature increased. At high temperatures, the NO to NO_2 oxidation reaction is counteracted by the reduction reaction as demonstrated by Djéga-Mariadassou et al. [101] in mixture with propene (Fig. 5.8).

5.5 NO_x Removal by Nonthermal Plasma and Energy Cost

5.5.1 Effect of Hydrocarbons

The NO to NO_2 conversion significantly depends on gaseous mixtures. As shown in the previous section, the hydrocarbon addition leads to significantly influence the NO_x chemistry during plasma remediation leading to the promotion of the oxidation of NO to NO_2 and lower the energy cost of this oxidation. According to the calculations of Park et al. [92], the initial HC concentration more than 500 ppm (except CH_4), and O_2 content more than 10%, almost does not affect the NO oxidation to NO_2. Experimental work of Khacef et al. [22] on the NO_x remediation in the presence of propene resulted in the similar conclusion as shown in Fig. 5.9.

Figure 5.10 shows the NO and NO_x (NO + NO_2) conversion rate as a function of SIE for the cases without propene and with 500 ppm propene. In the absence of hydrocarbons, less than 55% of the initial NO is converted to NO_2 even for the highest SIE, and the NO_x removal was zero. When 500 ppm of propene is added to the gas stream, the oxidation of NO to NO_2 was enhanced due to peroxy radicals HO_2 (see previous section).

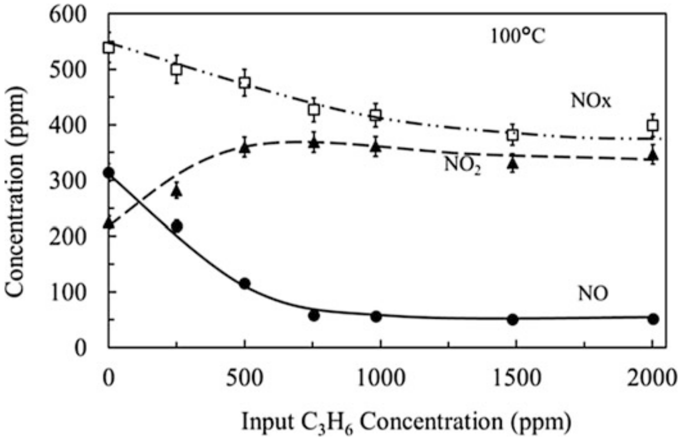

Fig. 5.9 Effect of inlet propene concentration on NO to NO_2 conversion in plasma processing of 500 ppm NO in air ($T = 100$ °C, SIE = 44 J/L) [22]

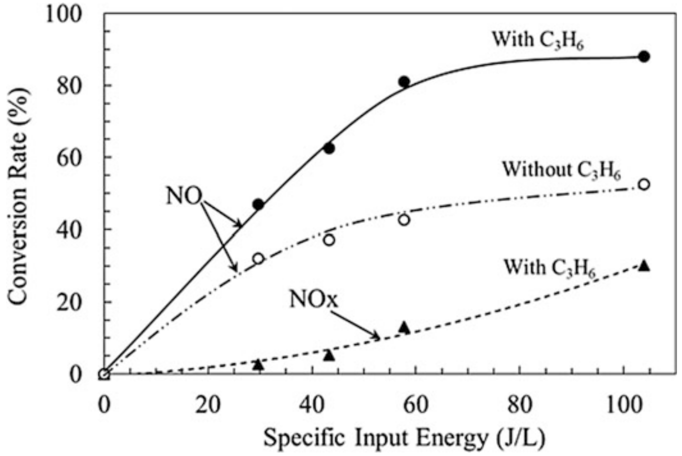

Fig. 5.10 Effect of SIE on NO and NO_x conversion without and with 500 ppm propene in NO (500 ppm)-air (195 mJ/pulse and repetition rate up to 40 Hz) [22]

5.5.2 *Energy Cost*

One of the relevant parameters in the cleaning of pollutant gas (NO_x, VOCs, etc.) is the energy cost for removing the unwanted molecules. To be competitive with other technologies, the nonthermal plasma technology applied for cleaning industrial or automotive exhaust gases should be the less consuming energy. The energy cost of the plasma-chemical processes is closely related to its mechanisms, and the same plasma processes in different discharge systems or under different conditions result in entirely different expenses of energy. Usually, the plasma processing literature present the fraction of NO_x removed as a function of parameters such as the specific input energy (SIE = mean input power/flow rate), the residence time of the gas into the reactor, or the applied voltage to the plasma reactor. SIE is the parameter commonly used to evaluate the NO_x conversion efficiency in the plasma. However, if this parameter is important to characterize the electrical energy consumption of the process, one should take into account the way to achieve the chosen energy value. It means that for a given plasma reactor, the plasma chemistry strongly depends on the energy transfer from the power source to plasma reactor. Many parameters, such as type of discharge (pulse, AC), high-voltage characteristics (amplitude, rise time, duration, frequency), electrode configuration, and efficiency of chemical reactions, have to be taken into account to optimize the process for a better energy efficiency.

The lowering of energy cost for NO_x conversion was obtained by many research groups for different systems. Niessen et al. [88] examine the effect of ethylene on the conversion of NO_x in a DBD. They reported an improvement of NO removal efficiency by an order of magnitude in the presence of 2000 ppm C_2H_4. Without C_2H_4 in the gas mixture, 48 eV/molecule (experiment) and 61 eV/molecule (model) are needed for the removal of one NO molecule at 90% removal rate, while adding 2000 ppm C_2H_4 leads to energetic cost of only 6 eV/molecule (experiment) and 9.6 eV/molecule (model).

In pulsed DBD experiments with gas mixture simulated diesel exhaust, Khacef et al. [22, 102] obtained a change of the energy cost for NO removal with temperature in the presence of C_3H_6. The authors demonstrate that, for a given reactor under the same gas composition and equivalent SIE, the NO removal efficiency is optimum for a low input energy per pulse and a high discharge frequency as shown in Fig. 5.11. The modeling of Filimonova and Amirov [103] on the removal of toxic impurities from air flow by a pulsed corona discharge resulted in similar conclusion. These authors confirm that it is possible to increase the removal efficiency at a constant total energy deposition by choosing the optimum pulse repetition rate and the energy deposited per discharge pulse. As example, for a SIE of about 27 J/L, NO conversion is more than 90% for 35 mJ/pulse and 200 Hz and less than 30% for 195 mJ/pulse and 30 Hz.

Whatever the pulsed discharge operations used, the main end products of the process are NO_2, CO, CO_2, CH_2O, CH_3ONO_2, CH_2O_2, HONO, and $HONO_2$. However, marked differences in final concentrations of these species were observed according the operating conditions.

Figure 5.12 shows an example of NO_x concentrations measured at the exit of a pulsed DBD reactor as a function of SIE in dry and humid (10% H_2O) gas mixture at 260 °C. With increasing energy deposition, the NO conversion and NO_2 formation efficiencies have been improved by increasing the gas temperature (up to 260 °C) and by adding 10% H_2O in the dry gas mixture. As example, the energy deposition of only 7 J/L was required to achieve maximum NO to NO_2 oxidation efficiency (up to 92%). This efficiency rate was only 61% when no water is present in the gas stream.

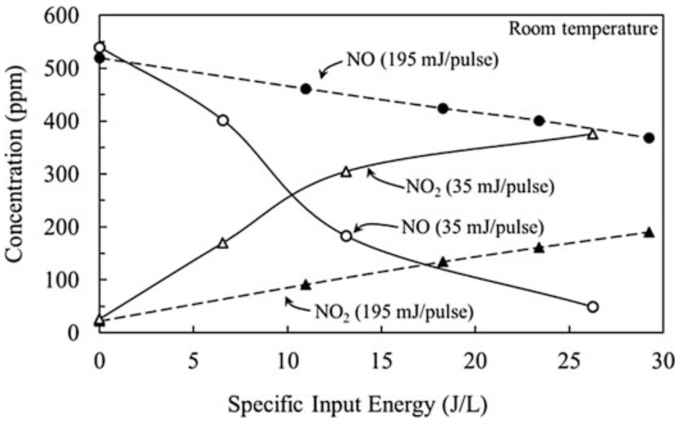

Fig. 5.11 NO and NO_2 concentrations as a function of SIE for pulse energy of 35 and 195 mJ. Gas mixture: O_2 (10%)-NO (500 ppm)-C_3H_6 (500 ppm)-N_2 (balance) [102]

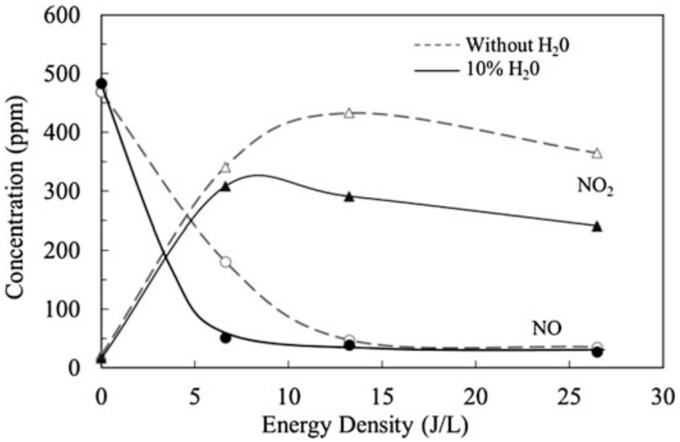

Fig. 5.12 Effect of SIE on NO_x conversion at 260 °C. Gas mixture: O_2 (10%)-NO (500 ppm)-C_3H_6 (500 ppm)-N_2 (balance) without H_2O and with 10% of H_2O [22]

The energy cost for humid plasma-induced NO removal is dramatically reduced to values scaling from $\approx$ 15 eV/molecule at 27 J/L down to $\approx$ 4 eV/molecule at 7 J/L. Bröer et al. [104] investigated synthetic gas mixture containing higher O_2 concentration (18%) with C_2H_4 instead of C_3H_6 and found that the maximum NO removal rate was 92% with an energy cost around 100 eV/molecule. In the modeling of the NO oxidation process in simulated exhaust containing O_2-H_2O-CO_2-CO-NO-H_2-C_3H_6-C_3H_8-N_2, Dorai and Kushner [90] observed that at 58 J/L, the energy cost decreases from 240 eV/molecule for a single pulse to 185 eV/molecule when the same energy was distributed over 20 pulses.

For vehicle exhaust systems, it was established that the observed chemistry in the plasma includes the conversion of NO to NO_2 as well as the partial oxidation of hydrocarbons. The presence of the unburned hydrocarbons (UHCs) in the exhaust is very important for the plasma- catalytic $deNO_x$ process for essentially four reasons [1, 2, 22, 50, 87–89, 95].

(a) UHCs enhance the gas-phase oxidation of NO to NO_2 and lower the energy cost of this oxidation.
(b) The partial oxidation of UHCs leads to produce chemical species such as aldehydes and alcohols useful for the catalytic reduction of NO_x. For some catalysts, the partially oxygenated hydrocarbons are much more effective compared to original hydrocarbons in reducing NO_x to N_2.
(c) The UHCs minimize the formation of acid products.
(d) UHCs prevent the oxidation of SO_2 thus making the plasma-catalytic process tolerant to the sulfur content of the fuel.

5.6 Plasma-Catalytic Process for NO_x Abatement

5.6.1 Why Do We Need Plasma for NO_x Abatement in Industrial Processes

As already discussed in the literature, NO_x trap systems and assisted NO_x reduction using SCR catalysts are commonly the two industrial processes developed in order to reach the limitation in terms of NO_x emission [5–8]. An alternative solution, not industrialized yet, is the use of hydrocarbons available in the exhaust, as reductant, to reduce the NO_x. However, in order to reach low temperature NO_x abatement, no catalytic system exists nowadays; thus, the combination of plasma and catalytic system would be a solution.

5.6.2 New Processes and Industrial Technologies

Atmospheric pressure NTP hybrid exhaust aftertreatment systems in the absence of noble metals has been developed by Kuwahara et al. [105]. Two types of new environmental protection systems (a dry system and a wet system), which enable the production of ultralow CO_2, particulate matter, and NO_x emissions as well as reduced fuel consumption and low cost, are investigated for diesel engines, marine engines, and combustion boiler applications. This paper reports the principles of the dry system and some recent experimental results of laboratory tests. The NO_x reduction comprises three flow processes: (i) adsorption, (ii) heating, and (iii) cooling processes. The heating process corresponds to the regeneration process. This dry system demonstrates excellent energy efficiencies that meet Japanese national regulations regarding automobile diesel engine exhaust gas. In this study, approximately 60% of NO_x conversion in the exhaust was achieved after 35 h on stream. An energy efficiency of about 143 g $(NO_2)/^{kWh}$ was then achieved for NO_x reduction.

Recently, two types of innovative NTP environmental protection systems [106, 107] using non-noble metals and which enable the production of ultralow CO_2, particulate matter (PM), and NO_x emissions as well as reduced fuel consumption and cost were proposed. These NTP systems are realized for diesel hybrid engines, marine engines, and combustion boiler applications. In these systems, the plasma technology is combined with other environmental technologies such as adsorption or wet-type chemical scrubbers. These technologies are expected to be new global environmental protection technologies, as they will enable an avoidance of the difficulties in catalytic methods and ordinary plasma methods. Other systems using a combination of NTP process and normal EGR (exhaust gas recirculation) led to similar results [108, 109].

5.6.3 Lab-Scale and Fundamental Studies

5.6.3.1 SCR of NO_x by Ammonia Assisted by Plasma

Only few studies dealt with selective catalytic reduction of NO_x by ammonia assisted by plasma. Thus, the SCR of nitrogen oxides by a combination of DBD plasma and a monolithic V_2O_5-WO_3/TiO_2 catalyst using ammonia between 100 and 250 °C was proposed [30]. Figure 5.13 summarizes the results obtained in that study. The gas mixtures used were representative of diesel exhaust gases. For gas mixtures in which 95% of the nitrogen oxides were in the form of NO, the removal of NO_x below 150 °C in the absence of plasma was negligible. Using the catalytic bed in the post-discharge DBD, about 70% of the NO_x was reduced at temperatures as low as 100 °C. Due to the coexistence of NO and NO_2 on the catalyst, the selective catalytic reduction was enhanced. Similar effects were observed for the SCR in gas mixtures containing equal amounts of NO and NO_2 without plasma treatment. This study clearly shows the efficiency of the plasma catalyst system at temperatures lower than those used for the catalytic system alone.

The potential of plasma-enhanced SCR for NO_x removal from exhaust gases containing high concentration of oxygen was investigated in a combination of dielectric barrier discharges with a monolithic V_2O_5-WO_3/TiO_2 catalyst using ammonia as a reducing agent. Experiments were performed which showed the temperature dependence of NO_x removal from synthetic gas mixtures, for very low temperatures (between 100 and 250 °C). It was shown that for $T < 200$ °C, the NO reduction rate decreased sharply as long as NO_2 concentration was low compared to NO concentration. This was the case when no plasma treatment was performed prior to catalytic treatment. With plasma treatment even at temperatures

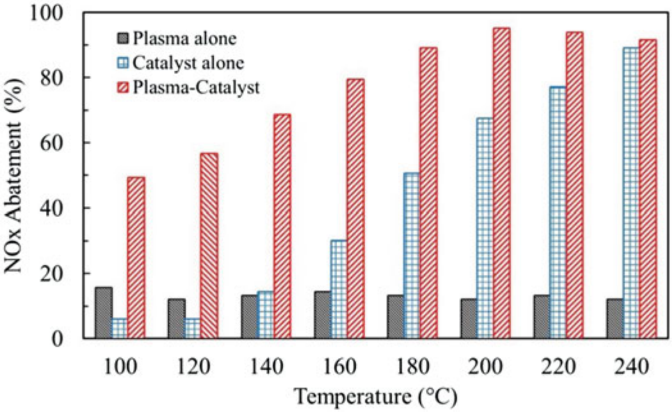

Fig. 5.13 DBD plasma-enhanced NH_3-SCR of NO_x (V_2O_5-WO_3/TiO_2 catalyst, synthetic gas mixture O_2 (13%)-H_2O (5%)-N_2 (82%)-NO_x (500 ppm)-NH_3 (500 ppm) [30]

as low as 100 °C, NO_x reduction reached values up to 70%. An explanation of these results could be given by experiments with gas mixtures containing NO_2 concentration, which were nearly equal to the NO concentration: without plasma treatment at 100 °C, the degree of catalytic NO_x reduction was 70%, also. From this result, it is concluded that on a V_2O_5-WO_3/TiO_2 catalyst at low temperatures, NO can be reduced in the presence of NO_2. Similar kinetic behavior was found in reactions of NO_x with zeolite-fixed ammonium ions [110].

In summary, one can conclude that the pre-treatment of the exhaust gas by nonthermal plasma enhances selective catalytic reduction rates at temperatures below 200 °C by oxidation of NO to NO_2, which was already proposed by McLarnon and Penetrante [75].

5.6.3.2 SCR of NO_x by Hydrocarbons Assisted by Plasma

Published literature reports on the application of the plasma-assisted HC/SCR to the lean-NO_x catalysis have focused primarily on the in situ activation by nonthermal plasma of the gas-phase reactants such as NO and/or HCs leading to NO_2 and oxygenated species [101, 111, 112]. In these studies, alumina- or zeolite-based catalysts were used coupled with NTP DBD in the wide temperature range (100–500 °C). The results clearly show that in the presence of the plasma, the catalytic activity is shifted to low temperature with high conversion of NO, and industrial application was clearly shown [110].

5.7 Role and Beneficial Effect of Plasma

5.7.1 Importance of NO_2 in the Catalytic Reduction of NO_x

Djéga-Mariadassou and co-workers showed that a three-function catalyst model for SCR NO_x with hydrocarbons could be proposed in the absence of plasma [101, 112, 113]. That model is based on experimental evidence for each function, during temperature programmed surface reactions (TPSR), and has been verified during stationary experiments. A general scheme of the model is shown in Fig. 5.14. The first function F1 leads to the oxidation of NO to NO_2. The second function F2 is the mild oxidation of hydrocarbons through organic nitrogen-containing intermediates (R-NO_x) leading to $C_xH_yO_z$ intermediate species such as aldehydes or alcohols. The third function F3 involves the NO reduction by subsequent formation of N2 assisted by the oxidation of reductants to CO_2/H_2O over transition metal cations. The previous $C_xH_yO_z$ intermediate species can achieve their own total oxidation by cleaning the adsorbed oxygen species left by NO dissociation.

It is very difficult to find the best design of the catalyst to simultaneously initiate the three functions by itself. Thus, an external device can be developed to substitute functions F1 and F2, providing the catalyst the appropriate oxygenated species, in the full range of temperature. For the vehicle exhaust systems, it was established that the plasma chemistry includes the conversion of NO to NO_2 as well as the partial oxidation of hydrocarbons. The presence of the unburned hydrocarbons in the exhaust is very important for the plasma-catalytic deNO_x process for multiple reasons. First, unburned hydrocarbons enhance the gas-phase oxidation of NO to NO_2 and lower the energy cost for this oxidation. Secondly, their partial oxidation leads to produce, in the whole range of reaction temperature, chemical species such as aldehydes, alcohols, and R-NO_x useful for the catalytic reduction of NO_x. For some catalysts, the partially oxygenated hydrocarbons are much more effective compared to original hydrocarbons in reducing NO_x to N_2. Thirdly, unburned hydrocarbons prevent the oxidation of SO_2 thus making the plasma- catalytic process tolerant to the sulfur content of the fuel. These "intermediate" species are needed for function F1 and F2 and, furthermore, for the third function itself. The NTP could substitute for the first two functions (F1, F2) of the catalysts as already proposed elsewhere [30, 101, 105–109, 112] (Fig. 5.15).

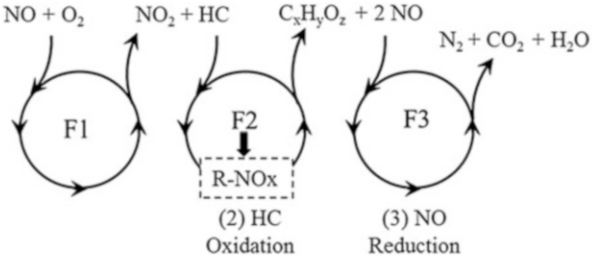

Fig. 5.14 Scheme for the three-function catalyst: concerted actions for the selective reduction of NO by hydrocarbons ($C_xH_yO_z$, partially oxidized HC)

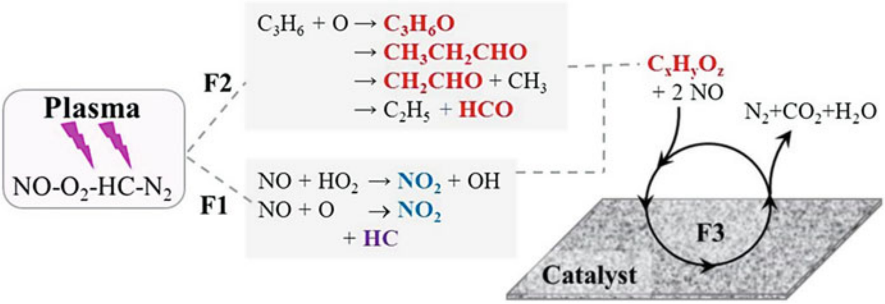

Fig. 5.15 Three-function mechanism adapted for plasma-catalytic processes

This result demonstrates what can be the role of plasma in nonthermal plasma-assisted catalytic NO_x remediation. Furthermore, plasma is able to provide both NO_2 and $C_xH_yO_z$ intermediate species at low temperature. However, the behavior of the R-NO_x species formed in the plasma still remains to be studied. The NO_x removal leads mainly to N_2 as reported previously [114].

5.7.2 *On the Effect of the Catalysts in the Coupled Process*

Miessner et al. [34] have shown that the combination of HC/HCR and nonthermal plasma enhances the overall reaction and allows an effective removal of NO_x at relatively low temperature (Fig. 5.16). The oxidative potential of an NTP in off-gases with excess oxygen results in an effective conversion of NO to NO_2 that can be converted synergistically by HC/HCR to molecular nitrogen with appropriate catalysts. The hydrocarbon added has two essential functions: first, it assists the gas-phase oxidation of NO to NO_2 by the electric discharge in excess oxygen and, secondly, it reacts with NO_2 in the hydrocarbon SCR. Besides CO_2 and CO, significant amounts of formaldehyde and acetaldehyde are formed in the plasma-initiated gas-phase reaction. These and other by-products are involved together with the remaining propene in the subsequent catalytic reaction. With a specific input energy density lower than 15 Wh/m^3, a temperature of 300 °C, and a space velocity of 20,000 h^{-1}, NO_x conversions higher than 50% are obtained. The synergistic combination of NTPs and HC/HCR has been verified under real conditions with exhaust gas from a diesel engine.

The catalyst support itself, Al_2O_3, seems to be an appropriate catalyst for the reaction [34, 114, 115], and some modifications of Al_2O_3 and ZrO_2 were found to be effective as catalysts in this reaction. The role of plasma processing on NO_x reduction over alumina and basic zeolite NaY was examined by Cho et al. [111] and compared to a conventional NH_3/SCR system, and the results are comparable at low temperature (Fig. 5.17).

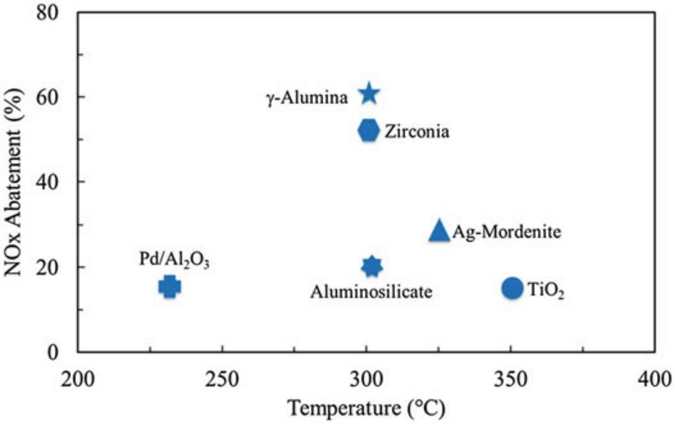

Fig. 5.16 Efficiency of various catalysts in the plasma-catalytic HC/HCR of NO_x (500 ppm NO and 1000 ppm C_3H_6 in N_2/O_2 (13%); SIE = 14 Wh/m) [34]

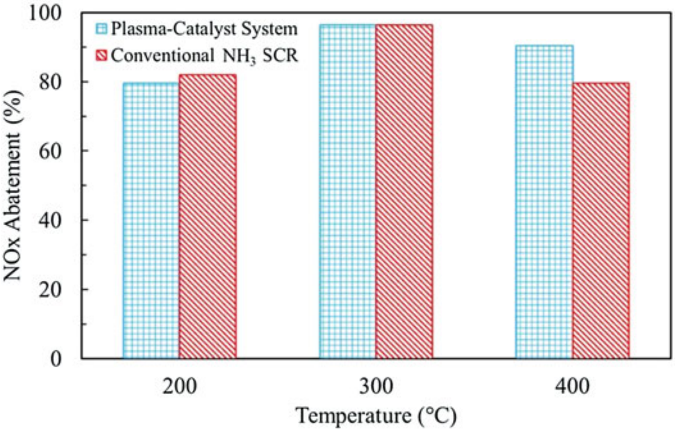

Fig. 5.17 Plasma-catalytic system and conventional NH_3/SCR in engine dyno tests as a function of temperature [111]

During the plasma treatment, NO is oxidized to NO_2, and propylene is partially oxidized to CO, CO_2, acetaldehyde, and formaldehyde. With plasma treatment, NO as the NO_x gas, and a NaY catalyst, the maximum NO_x conversion was 70% between 180 and 230 °C. The activity decreased at higher and lower temperatures. As high as 80%, NO_x removal over alumina was measured. For both catalysts, a simultaneous decrease in NO_x and aldehyde compound concentrations was observed, which suggests that aldehydes may be important components for NO_x reduction in plasma-treated exhaust, which was already proposed by Djéga-Mariadassou et al. [101, 112, 113, 116]. The authors proposed a simplified reaction mechanism to explain the enhanced deNO$_x$ activity induced by air plasma as follows: ozone production, NO and HC oxidation by ozone, and NO$_x$ reduction assisted by oxidized hydrocarbons to form N_2, H_2O, and CO_2. That later reaction proceeds on BaY catalyst at low temperatures and on CuCoY catalyst at high temperatures as evidenced by Schmieg et al. [115] and Lee et al. [117].

5.8 Conclusion

NTP catalytic processes are in development in order to reduce the NO$_x$ in the exhaust gases from industrial processes such as burners for stationary sources or in the exhaust line for mobile applications. It is clear that the plasma is able to create at low temperature the needed species for NO$_x$ abatement. Some coupled processes have been already tested at the pilot scale, but their industrialization will be possible only if the energy due to the plasma makes this process a low-cost process versus the existing ones. Another possible way to use the plasma to reduce NO$_x$ emission is the use of this one in the combustion process to avoid the NO$_x$ formation.

Thus, Lee et al. [118] have recently presented a new combustion technology that can reduce NO$_x$ emissions within industrial burners to single-digit parts per million levels without employing exhaust gas recirculation or other NO$_x$ reduction mechanisms. This new technology uses a simple modification of commercial burners, such that they are able to perform plasma- assisted staged combustion without altering the outer configuration of the commercial reference burner. Because this plasma burner acted as a low NO$_x$ burner and was able to reduce NO$_x$ by more than half compared to the commercial reference burner, this methodology offers important cost-effective possibilities for NO$_x$ reduction in industrial applications at source.

Acknowledgments The authors appreciate the considerable contribution of Professor Gérald Djéga-Mariadassou and Doctor Francois Baudin as well as engineers from Renault SA and PSA Peugeot-Citröen for their fruitful discussions on deNO$_x$ processes and engine chemistry.

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