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Studies of atmospheric-pressure microwave plasmas used for gas processing

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Abstract. This paper concerns the atmospheric-pressure microwave plasmas and their applications, mainly for gas processing. Several types of atmospheric-pressure microwave sources (MPSs), i.e. surface-wave-sustained MPS, nozzle-type MPSs, nozzleless MPSs, plasma-sheet MPSs and microwave microplasma sources – MmPSs (antenna- and coaxial-line-based) as well as their performance are presented. The presented experimental results on the optimization of selected MPSs are confronted with results of the modelling of the electromagnetic field in them. The paper deals also with the applications of MPSs for the processing of gases. Two types of the plasma gas processing were experimentally tested: decomposition of volatile organic compounds (VOCs) and reforming of VOCs (mainly methane) into hydrogen. Results of the laboratory experiments on the plasma processing of several highly-concentrated (up to 100%) VOCs, including freon-type refrigerants, in the waveguide-supplied MPSs showed that the microwave discharge plasma is capable of fully decomposing the VOCs at relatively low energy cost. The use of waveguide-supplied coaxial-line-based and metal-cylinder-based nozzleless MPSs to methane reforming into hydrogen turned out to be energetically efficient. These selected results show MPSs to be an attractive tool for gas processing, including the harmful gas decomposition and production of useful gases.

Key words: microwaves • plasma • volatile organic compounds • hydrogen

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Introduction

The emission of SO_2, NO_x (NO, NO_2), CO, CO_2, VOCs and other gaseous pollutants to the atmosphere influences heavily our environment. Therefore, efficient methods for the control and reduction of gaseous pollutant emission are strongly required. Nowadays, conventional methods, e.g. adsorption, absorption, catalytic combustion seem not to be efficient enough. Recently, potential of the atmospheric pressure thermodynamic equilibrium plasmas (called thermal plasmas), such as direct current (DC) torches from plasmatrons, radio frequency (RF) torches, and non-thermodynamic equilibrium plasmas (non-thermal plasmas), e.g. electron-beam generated plasmas, DC and pulsed corona discharges, corona discharges in packed-bed reactors, dielectric barrier discharges, surface discharges, microwave discharges, have been developed and tested for the processing of gaseous pollutants. The wide range of the plasma parameters, including gas temperature, offered by thermal and non-thermal plasmas, makes both plasmas applicable for the processing of various gases.

Some of the non-thermal plasma methods have been proved to be efficient for the destruction of gaseous pollutants (SO_x, NO_x, VOCs) of relatively low concentration [up to 1000 ppm (i.e. 0.1%)] in the working gas (e.g. [2, 5–7, 9, 24, 36, 37]). On the other hand, it has been found that the microwave non-thermal plasmas have the potential for efficient processing of gaseous pollutants (mainly VOCs) of relatively high concentration (up to 100%) in the working gas [11, 17, 20, 21, 29, 36]. Recently, the non-thermal microwave plasma has efficiently been used not only for the harmful gas decomposition but also for production of hydrogen by means of the plasma reforming of methane [15, 16].

In this paper we deal with the microwave plasmas classified as the atmospheric pressure non-thermal plasmas at elevated working gas temperatures (~ 4000 K), which can be used for gas processing. These temperatures are too low to form the local thermodynamic equilibrium (LTE) of the plasma, however they can be useful for the processing of stable molecules, for example for decomposing VOCs.

A short review of various atmospheric-pressure microwave sources (MPSs) and plasmas will be presented and their characteristics described in this paper. Designs, working conditions and plasma properties of several types of the MPSs, which have the potential for gas processing [11, 13, 15–17, 20–22, 29, 31, 36] will be presented. As examples, we will show results of the study of decomposition of a highly-concentrated VOC (freon HFC-134a) and of the reforming of methane CH_4 to produce hydrogen H_2 with the use of a waveguide-supplied coaxial-line-based nozzleless MPS and waveguide-supplied metal-cylinder-based nozzleless MPS.

Atmospheric-pressure microwave sources and plasmas

The microwave plasmas operating at atmospheric pressure can be induced by several types of microwave field applicators, which may be classified as follows:

A. Surface-wave-discharge MPSs:
   a) coaxial-line-supplied, called surfatrons (not presented in this paper),
   b) waveguide-supplied, called surfaguides.

B. Nozzle-type MPSs:
   a) coaxial-line-supplied coaxial-line-based (low gas flow rate, several l/min),
   b) waveguide-supplied coaxial-line-based (low and high (gas swirl, several hundred l/min) flow rates).

C. Nozzleless MPSs:
   a) waveguide-supplied coaxial-line-based (with or without an inner dielectric tube),
   b) waveguide-supplied metal-cylinder-based (with or without an inner dielectric tube),
   c) waveguide-supplied resonant-cavity-based.

D. Plasma-sheet MPSs:
   a) coaxial-line-supplied strip-line-based,
   b) waveguide-supplied.

E. Microwave microplasma sources – MmPSs (antenna- and coaxial-line-based).

F. Inductively coupled MPSs (not presented).

The above listed MPSs are known under different names, usually given by their inventors. The classification introduced by us is mainly based on the way of supplying the microwave energy (e.g., waveguide-supplied – supplied through a waveguide) and on the microwave principle of operation of the MPS (e.g., coaxial-line-based – the operation is based on the coaxial transmission line structure). In the name of MPS also its specific feature, e.g., nozzle-type (when a nozzle is an essential part of the MPS), is included.

Atmospheric pressure microwave plasmas generated in MPSs are often demanded to be sustained within a dielectric (e.g., fused silica) tube due to the chemical reactivity of processed gases. This can cause some problems when large power densities are deposited into the plasma, resulting in deterioration of the tube due to plasma-tube interactions. A solution to these problems is a high flow of the operating gas through the dielectric tube, which can convey the produced heat. Another solution is using an additional gas flow, called a gas swirl, together with the main gas stream to protect the walls from heat deterioration. Using either the high working gas flow rate or additional gas swirl weakens the microwave power limitations imposed on the MPSs used for the gas treatment.

Depending on the kind of MPS, the microwave power is supplied from a microwave generator (magnetron) to the MPS through either a coaxial cable or a rectangular waveguide.

Waveguide-supplied surface-wave-discharge MPS (surfaguide)

The waveguide-supplied surface-wave-discharge MPS is shown in Fig. 1. Microwave (2.45 GHz) power of about a few hundred watts is supplied from a magnetron via a standard WR-430 rectangular waveguide with a reduced-height section [21]. The discharge in the form of a plasma column is generated inside a quartz tube. The impedance-matching between the MPS and the microwave supplying system is achieved by the use of a tuning element in the form of a movable plunger placed at the end of the waveguide. The movable plunger enables minimizing the reflected microwave power.

This type of MPS was extensively studied (e.g., [22, 31]). It has been commercially developed for the abatement of fluorinated etching gases in the semiconductor industry [13].

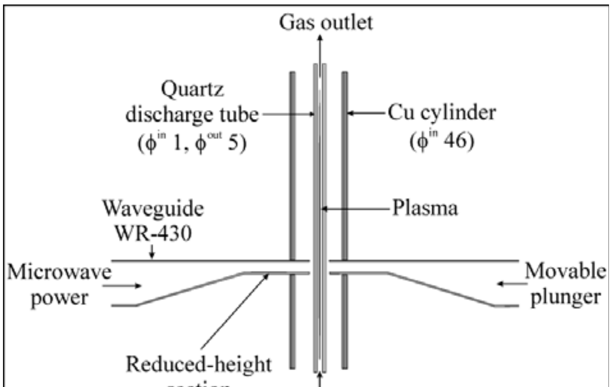

Fig. 1. The waveguide-supplied surface-wave-discharge MPS.

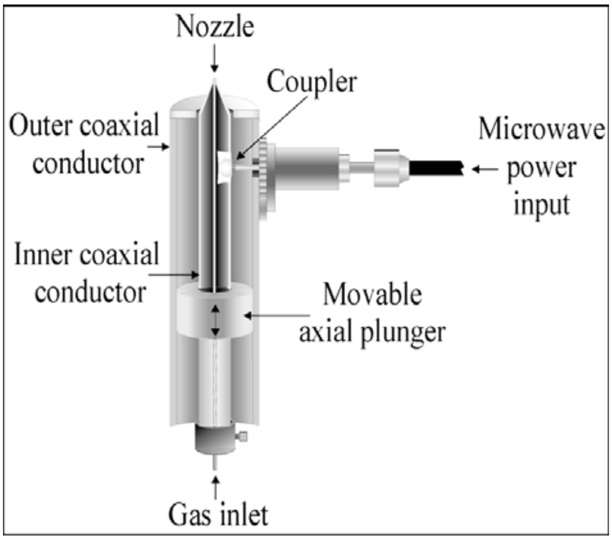

Fig. 2. The coaxial-line-supplied coaxial-line-based nozzle--type MPS.

Coaxial-line-supplied coaxial-line-based nozzle-type MPS

The nozzle-type MPSs first appeared as structures based on microwave coaxial line components (e.g., [29]). In these coaxial-line-supplied coaxial-line-based nozzle--type MPSs the microwave plasmas are induced in the form of a plasma “flame”, called also a plasma plume, at the open end of a rigid coaxial line, at the tip of its inner conductor with a conical nozzle (nozzle diameter about 1 mm) (Fig. 2). The use of a conical-nozzle inner electrode results in the microwave electric field around it high enough to initialize and sustain the plasma flame. The operating gas is delivered to the nozzle through the inner pipe (low gas flow rate, a few l/min) or several inner pipes (high gas flow rate, a few hundred l/min) made in the inner conductor. The power-handling capability of coaxial-line-based MPSs is generally limited to much less than 0.5 kW due to the low thermal strength of the coaxial line components, mainly coaxial line cable supplying microwaves. This type of MPS was used by us for destruction of fluorinated hydrocarbons (e.g., [29]).

Waveguide-supplied coaxial-line-based nozzle-type MPS

Concurrently with the coaxial-line-supplied coaxial--line-based nozzle-type MPSs the so-called waveguide--supplied coaxial-line-based nozzle-type MPSs have been developed (e.g., [20, 21, 29]). In these applicators the microwave plasma is also induced in the form of a plasma flame at the tip of a field-shaping structure that is similar to that of the coaxial-line-supplied coaxial--line-based nozzle-type MPSs. However, the microwave power is fed into this structure through a waveguide (usually rectangular), with a reduced-height section to enhance the electric field in the field shaping structure.

Two types of the waveguide-supplied coaxial-line--based nozzle-type MPSs have been developed, i.e. the waveguide-supplied nozzle-type MPS with a gas supply through the pipe(s) in the nozzle [20, 21, 29] (Fig. 3), and the waveguide-supplied nozzle-type MPS with a gas swirl around the nozzle (Fig. 4).

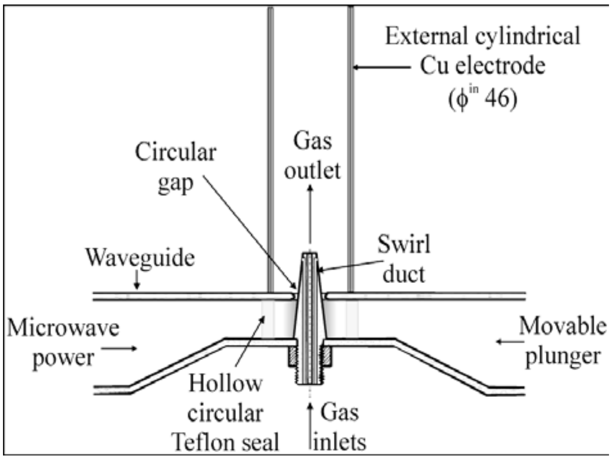

Fig. 3. The waveguide-supplied coaxial-line-based nozzle-type MPS operated at low or high gas flow rate.

In the waveguide-supplied coaxial-line-based nozzle-type MPS operated at low gas flow rate (a few l/min), shown in Fig. 3, the microwave (2.45 GHz) power of about a few hundred watts is supplied from a magnetron via a standard WR-430 rectangular waveguide to the field-shaping structure having the form of a conductor with a conical nozzle. The discharge in the form of a plasma flame is generated inside a quartz tube (not shown) directly above the metal conical nozzle. The waveguide-supplied nozzle-type MPS operated at low gas flow rate was used by us for destruction of various VOCs, including freon-type refrigerants [20, 21, 29].

The sketch of the waveguide-supplied coaxial-line--based nozzle-type MPS operated at high gas flow rate (a several hundred l/min) is shown in Fig. 4. In this MPS, two flows are formed in a quartz cylinder placed coaxially with the nozzle: central flow (the processed gas) and an additional swirl flow (e.g., nitrogen). The processed gas flow is introduced centrally to the discharge zone through a conical nozzle. The swirl flow is supplied tangentially through the four inlets, creating a spiral vortex flow in the quartz cylinder. This stabilizes the discharge in the centre of the cylinder and protects

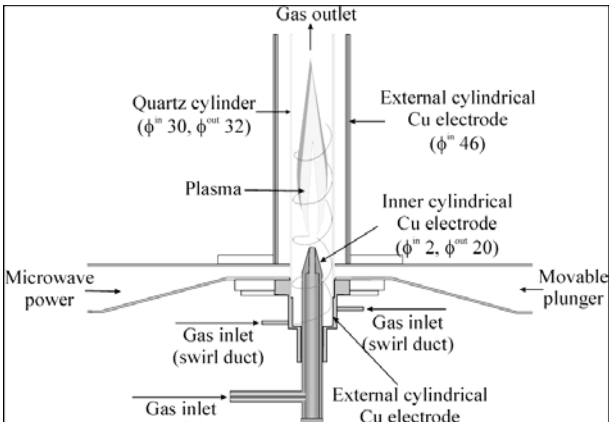

Fig. 4. The waveguide-supplied coaxial-line-based nozzle--type MPS operated at high gas flow rate due to an additional gas swirl.

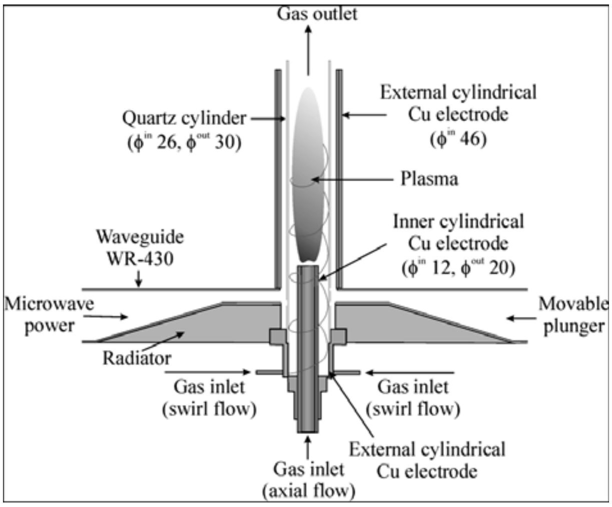

Fig. 5. The waveguide-supplied coaxial-line-based nozzleless MPS.

the wall from the discharge heat [36]. A disadvantage of both the presented waveguide-supplied coaxial-line--based nozzle-type MPSs is vulnerability of the nozzle to erosion. Such a disadvantage has been avoided in the waveguide-supplied coaxial-line-based nozzleless MPS presented below.

Waveguide-supplied coaxial-line-based nozzleless MPS

The sketch of the waveguide-supplied coaxial-line--based nozzleless MPS is presented in Fig. 5 [15–17]. This device is similar to the waveguide-supplied coaxial--line-based nozzle-type MPS operated at high gas flow presented in Fig. 4. However, in the MPS shown in Fig. 5 the nozzle electrode was replaced by a cylindrical hollow electrode (of an inner diameter of several mm). Changing the shape of the inner metal electrode resulted in lower erosion of the electrode. When the coaxial-line system in this MPS is properly optimized, the electric field is high enough to initialize and sustain the plasma above the inner cylindrical electrode. Although a quartz (or another dielectric) tube is employed in the MPS, shown in Fig. 5, the waveguide-supplied coaxial-line--based nozzleless MPSs can work without it.

Waveguide-supplied metal-cylinder-based nozzleless MPS

The design of the waveguide-supplied metal-cylinder--based nozzleless MPS is shown in Fig. 6. In this case there is not any inner electrode forming a coaxial-line system as is in the MPS presented in Fig. 5. When the MPS is properly optimized and a swirling gas is injected into the operation region, the plasma is generated inside a metal cylinder or in the quartz cylinder inserted in it, as shown in Fig. 6. The gas swirl stabilizes the plasma and also protects the cylinder wall (metal or quartz) from the discharge heat [15–17, 36].

The waveguide-supplied metal-cylinder-type nozzleless MPS showed stable operation at power levels from about 600 up to 6000 W, provided that the total gas flow is sufficiently large (from 30 up to several hundred l/min).

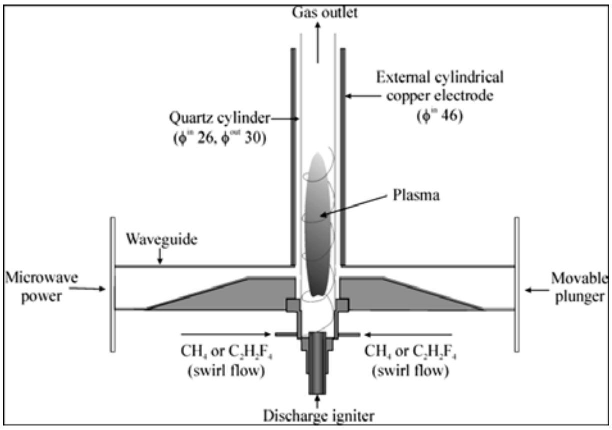

Fig. 6. The waveguide-supplied metal-cylinder-based nozzleless MPS.

The waveguide-supplied metal-cylinder-based nozzleless MPS was used by us for destruction of freon HFC-134a [17] and for producing hydrogen H_2 in the process of plasma reforming of methane CH_4 [15, 16].

To meet industrial requirements, the waveguide--supplied metal-cylinder-based nozzleless MPS, as well as other MPSs, require optimization with the goal of improving the power coupling efficiency, i.e. the efficiency of power transfer from electric field to plasma, and stability of the MPS operation. For that purpose, the three-dimensional (3-D) electric field distributions in the MPS were determined by us numerically employing COMSOL software. Using these results and a newly proposed two-port network method, the tuning characteristics of the MPS, employing a plunger as a tuning means, could be calculated. A good agreement was found between the tuning characteristics obtained experimentally and numerically. Then, optimization of the MPS was performed using these characteristics to ensure good power coupling and stability of the plasma source operation. Our numerical and two-port network methods were presented in detail in Ref. [32].

Waveguide-supplied resonant-cavity-based MPS

Recently, the waveguide-supplied resonant-cavity-based MPSs have regained interest and several designs have been presented [1, 3, 25–28, 35]. In general, in such MPSs the microwave power is delivered usually through a rectangular waveguide, to a microwave cavity (rectangular or cylindrical), that is designed to support the existence of a selected number of microwave resonant oscillations (modes). The microwave power, stored in the cavity, is capable of igniting and maintaining the plasma. Operating gas or gases are fed into the cavity either tangentially through several inlets or axially through a metallic nozzle or pipe, placed centrally in the cavity. The plasma is usually generated in a quartz tube acting as a plasma reactor.

In general, two types of the waveguide-supplied resonant-cavity-based MPS are employed. The first is a simple cavity terminating the microwave power supplying waveguide. The plasma is produced either in a quartz tube passing through the cavity axis or directly over the cavity exit (a hole or holes in the cavity cover). The second type of the waveguide-supplied resonant-

-cavity-based MPS is more complex. In some designs, the resonant cavity houses two opposite electrodes placed coaxially in the cavity axis. A gas to be treated flows into the cavity through a gas inlet, and passes between the electrodes, so that a microwave plasma is initiated between the two electrodes. One of the electrodes has an axial hole to provide an outlet from the resonant cavity for the plasma and operating gas. In other designs, the resonant cavity is equipped with a relatively short coaxial-line-like structure. The inner electrode of this structure, in the form of a metallic pipe, nozzle or rod with a conical tip, serves for the plasma ignition and delivers the operating gas to the cavity (nozzle or pipe case). This metallic insert in the cavity influences the resonant frequency of the cavity and can be used for tuning the cavity to the microwave generator frequency.

At present, our research on the use of a waveguide-supplied resonant-cavity-based MPS of the second type for production of hydrogen from CH_4 showed that CH_4 conversion degree, H_2 production rate and the corresponding energetic mass yield were 25%, 120 gH_2/h and 30 gH_2/kWh, respectively, at an absorbed power of 4000 W and CH_4 flow rate of 50 l/min. The MPS performance optimization is in progress.

Coaxial-line-supplied strip-line-based and waveguide-supplied plasma-sheet MPSs

Common forms of the plasmas delivered by MPSs operated at atmospheric pressure resemble flames or cylindrical columns. However, an atmospheric-pressure plasma delivered in the form of a plasma sheet would be very attractive for many applications (e.g., surface modifications, long-line welding).

We developed two MPSs that are capable of delivering argon plasma sheet at atmospheric pressure, based either on the strip-line [18] or on the rectangular waveguide structure [19].

Microwave microplasma sources (MmPSs)

There is a growing interest in microdischarge sources operating at atmospheric pressure [2]. We developed two types of microwave microplasma sources (MmPS): antenna- [30] and coaxial-line-based [12]. Our design of MmPS based on the coaxial line is shown in Fig. 7. The microwave (2.45 GHz) power is delivered to the plasma region via standard 50 Ω coaxial cable. Microplasma is generated at the tip of tungsten (or graphite) rod electrode. The MmPS exhibited a stable operation in Ne, Ar, N_2 and air at atmospheric pressure in the form of a small plasma flame at absorbed microwave powers of 6–80 W and gas flow rates of 0.5–25 l/min.

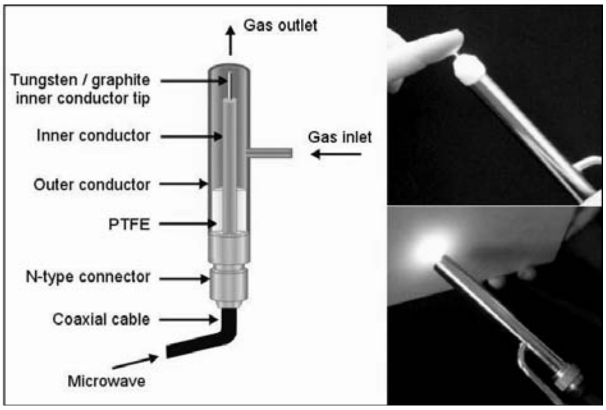

Fig. 7. The coaxial-line-supplied coaxial-line-based MmPs.

Gas processing by microwave plasmas – selected results

MPSs have found many applications, also for gas processing [23, 33, 34]. Below some selected examples of using the presented MPSs for decomposition of VOCs and hydrogen production are presented.

Decomposition of VOCs

Decomposition of several VOCs: aliphatic hydrocarbon (H-C) – methane CH_4, aromatic H-C – toluene $C_6H_5CH_3$, halogenated aliphatic H-Cs – carbon tetrachloride CCl_4 and chloroform $CHCl_3$, aromatic chlorofluorocarbons: CCl_3F (CFC-11) and CCl_2F_2 (CFC-12), hydrochlorofluorocarbon $CHClF_2$ (HCFC-22), hydrofluorocarbon $C_2H_2F_4$ (HFC-134a) and fluorocarbon C_2F_6 (CFC-116) in their mixtures with synthetic air or nitrogen in the atmospheric-pressure plasmas generated by the waveguide-supplied nozzle- and nozzleless-type MPSs was studied by us. The use of N_2 is recommended due to smaller problems with harmful by-products [17, 20, 21, 29]. In spite of using N_2, no toxic HCN or NH_3 were formed. In our investigations the concentrations of the processed VOCs were relatively large, i.e. even up to 100%.

When the waveguide-supplied nozzle-type MPS was used for CFC-11 decomposition, the removal rate and energetic mass yield of CFC-11 decomposition were 310 g (CFC-11)/h and 1000 g (CFC-11)/kWh, respectively (initial CFC-11 concentration – 50%, gas flow rate – 4 l/min, absorbed power – 4000 W) [21].

When the waveguide-supplied metal-cylinder-based nozzleless MPS was used, the decomposition rate and energetic mass yield of HFC-134a decomposition were 34 kg (HFC-134a)/h and 34 kg (HFC-134a)/kWh, respectively (HFC-134a initial concentration – 100%, HFC-134a flow rate – 200 l/min, absorbed – 1000 W).

The results of destruction of other harmful gases were in detail presented in [20, 21, 29].

Production of hydrogen via methane reforming

The reforming of methane CH_4 to produce hydrogen H_2 in the atmospheric-pressure plasma generated by the waveguide-supplied coaxial-line-based nozzleless MPS (Fig. 5) and the waveguide-supplied metal-cylinder-based nozzleless MPS (Fig. 6) were tested [15, 16].

The methane flow rate was up to 175 l/min. The absorbed microwave power could be changed from 1000 to 5000 W. The hydrogen production rate and the corresponding energetic mass yield were up to 110 g (H_2)/h and 75 g (H_2)/kWh, respectively, taking into account input energy from a plug. Soot formed as a by-product of methane pyrolysis did not influence the hydrogen production efficiency. The microwave plasma method presented by us is more efficient than other plasma methods (dielectric barrier discharge [10], gliding arc [8], plasmatron without catalyst [4]), and water electrolysis [14]).

Summary and conclusions

Several MPSs suitable for gas processing (e.g., decomposition of VOCs, hydrogen production via methane reforming) were presented in this paper.

It was found that the rate of decomposition of harmful gases (e.g., freons) and the corresponding energetic mass yield obtained using the waveguide-supplied MPS are superior to those when other plasma methods were employed for this purpose.

The energetic parameters of hydrogen production by the waveguide-supplied nozzleless-type MPS are attractive. The absence of oxygen compounds as by-products in the off-gas is highly beneficial. The proposed microwave plasma system for hydrogen production via methane reforming is expected to be of low cost and effective, and thus promising for industrial implementation.

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