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STUDY OF RF-DBD DISCHARGE IN AR/CH4 MIXTURE IN THE PRESENCE OF NICKEL FOAM AT LOW PRESSURE

Abstract

This paper investigates the effect of nickel foam on the structure and properties of Radio-Frequency Dielectric Barrier Discharge (RF-DBD) in an Ar/CH4 mixture at a low pressure of 0.5 Torr. Experiments were conducted by varying the supplied power, gas flow, and distance between the catalyst and the RF electrode. It has been shown that an increase in plasma power and a change in CH_4 gas flow in the presence of a catalyst leads to a noticeable shortening of the plasma glow length in a quartz tube. It was found that increasing the distance between the catalyst and the RF electrode reduces the ability to maintain plasma in the region downstream of the catalyst. The analysis of optical emission spectra revealed a decrease in the intensity of carbon-containing radicals, atomic and molecular hydrogen after the catalyst, which indicates its active participation in plasma-catalytic processes. Raman analysis confirmed the formation of amorphous carbon deposits on the nickel foam surface. It has been established that nickel foam not only modifies the structure of the RF-DBD discharge, but also significantly affects the distribution of active particles in the plasma, changing the conditions for plasma-catalytic reactions. The results obtained provide a deeper understanding of the mechanisms of interaction between low-temperature plasma and porous metal catalysts and can be used in the development of effective plasma-catalytic systems for the conversion of hydrocarbon gases.

Keywords: plasma catalysis, RF-DBD discharge, optical emission spectroscopy, catalyst, nickel foam.

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Introduction

Gas conversion of hydrocarbons, particularly CH_4, is one of the key industrial methods for producing hydrogen and is widely used in the chemical and energy industries (steam reforming, partial oxidation, dry reforming) [1]. Traditional gas conversion methods are based on thermal activation of molecules and require high temperatures, typically above 600-800°C, which is accompanied by

significant costs and CO_2 emissions [1]. Thermal gas conversion processes remain heavily dependent on fossil fuels and face limitations in terms of flexibility, selectivity, and decarbonization [2]. Increasing temperatures in traditional processes often leads to side reactions and catalyst degradation, complicating process control and reducing process stability [3].

The continuous growth of interest in alternative gas conversion methods has led to the active development of plasma technologies, in particular non-thermal plasma [4]. In non-thermal plasma, electrons have high energy (several eV), while the gas temperature remains close to room temperature, which in turn allows the excitation, dissociation, and ionization of molecules without heating the entire gas phase [1]. Consequently, plasma conversion makes it possible to carry out reactions that are thermodynamically limited at low temperatures in thermally activated processes [5].

Compared to other activation methods, such as thermal, photocatalytic and electrochemical conversion, non-thermal plasma provides direct energy supply to the electronic subsystem, rapid response to parameter changes, and the ability to operate at different pressures (including operation at atmospheric pressure), making it a promising tool for controlled gas conversion [6–10]. However, plasma itself often demonstrates limited selectivity, as the highly reactive environment promotes the formation of a wide range of products [2].

To increase selectivity and targeted control of chemical pathways, plasma processes are increasingly being combined with catalysts to form plasma-catalytic systems [3, 11, 12]. In non-thermal plasma, the catalyst can be placed directly in the discharge zone (in-plasma) or after the plasma (post-plasma). In the in-plasma mode, the catalyst is located directly in the discharge zone and interacts with ions, electrons, radicals, and excited particles, as well as being exposed to electric fields. This can lead to surface modification and changes in reaction kinetics. In post-plasma mode, the catalyst interacts mainly with long-lived neutral active particles and products of plasma chemical reactions. At the same time, the influence of electric fields and charged particles is significantly weakened. As a result, the activation mechanisms and selectivity of reactions in these configurations differ fundamentally [1, 12].

Active plasma particles, such as radicals, ions, and excited molecules, can interact with the catalyst surface, changing reaction pathways and lowering effective energy barriers [1, 3–5, 11, 12]. Metal catalysts can enhance catalytic reactions and, at the same time, have a reverse effect on plasma by altering the electric field distribution, electron density, and discharge characteristics [2]. The role of the catalyst in plasma-catalytic systems remains a subject of active debate, since in some cases it can lead to the quenching of plasma-activated radicals and a decrease in the overall reaction rate. In plasma-catalytic systems, metal catalysts based on Ni, Cu, Ag, Fe, as well as noble metals Pt, Pd, Rh, and Ru, including systems deposited on oxide supports such as Ni/Al_2O_3, Ni/SiO_2, as well as bimetallic and structured catalysts used for the conversion of CH_4 and other hydrocarbons [2].

Nickel catalysts are widely used in CH4 conversion reactions due to their high activity in breaking C-H bonds [3]. The use of nickel foam is of particular interest because its developed porous structure provides a high surface area and can enhance local plasma-catalytic interactions [1].

Plasma diagnostics play a key role in understanding the mechanisms of plasma-catalytic conversion [13]. Optical emission spectroscopy (OES) is widely used to identify excited atomic and molecular particles such as H, CH, and C_2 [14]. Raman spectroscopy is a key tool for analyzing carbon-containing solid products, including graphite-like and amorphous phases, which are formed in plasma CH_4 decomposition processes [13]. However, the influence of the spatial arrangement of the metal catalyst on the structure of the RF-DBD discharge and the distribution of active particles at reduced pressure remains insufficiently studied.

In this work, studies were conducted using nickel foam as a catalyst. The effect of the catalyst on CH_4 gas conversion in an RF-DBD discharge was studied depending on the power, catalyst location distance, and gas flow.

Materials and methods

The diagram of the experimental setup for studying RF-DBD plasma is shown in Figure 1. The plasma is ignited inside a horizontally positioned quartz tube by a radiofrequency (RF) generator (1)

with a frequency of 13.56 MHz, connected to a matching device (2). The outer diameter of the quartz tube (3) is 20 mm, and the wall thickness is 2 mm. In this configuration, the quartz discharge tube serves as the dielectric barrier separating the plasma from the powered RF electrode. Low pressure is provided by an Edwards XDS10-C UL fore-vacuum pump (4). Working gases, such as Ar and CH_4, are fed into the system through mass flow controllers (5). The high-frequency electrode (6) with an outer diameter of 4 mm is made of AISI 304 stainless steel. Copper tape (7) was selected as the grounding electrode. The pressure is controlled by an Edwards ADC Enhanced MKII controller sensor (8). Optical emission spectra were obtained using an Optosky ATL 30007 spectrometer (9). The obtained spectral lines were processed on a laptop (10).

In order to determine the optimal plasma (11) ignition parameter with nickel foam (12) with a thickness of 1.5 mm (Shenzhen Tianchenghe Technology Co. Ltd.), experiments were conducted at a pressure of 0.5 Torr depending on power, distance, and gas flow.

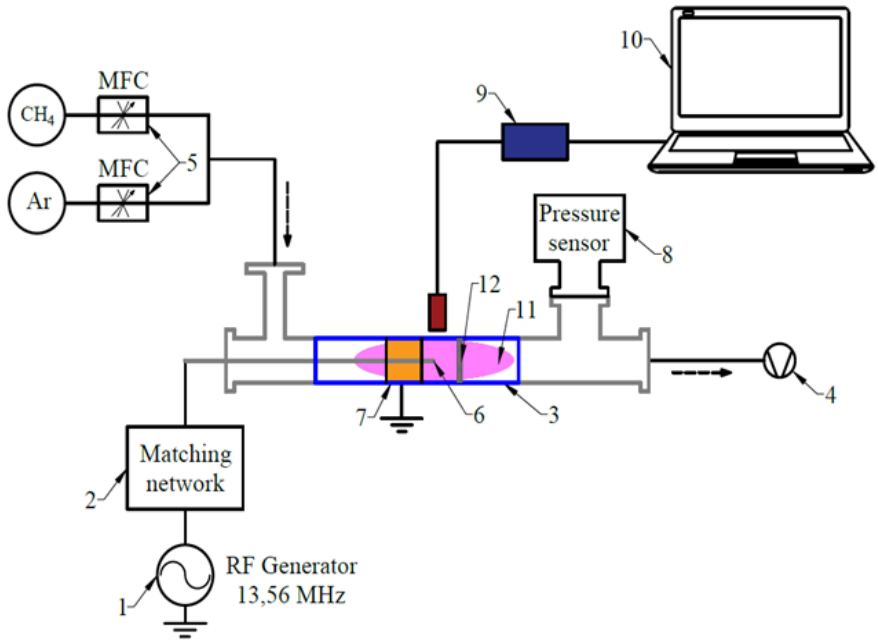

Figure 1 – Schematic diagram of the experimental setup for gas conversion:
1 – RF generator, 2 – matching device, 3 – quartz tube for gas discharge, 4 – fore-vacuum pump,
5 – gas mass flow controllers, 6 – RF electrode, 7 – grounding electrode, 8 – pressure sensor,
9 – optical emission spectrometer, 10 – laptop, 11 – plasma, 12 – catalyst (nickel foam)

Results and discussion

Photos of RF-DBD discharge with nickel foam at a constant flow of Ar (80 sccm) + CH_4 (25 sccm) gas and at a pressure of 0.5 Torr at different power values are shown in Figure 2. During the experiment, the catalyst was placed at a distance of 1 cm after the RF electrode. Figure 2 shows the following: as the discharge power increases from 11 W to 15 W, the length of the plasma glow along the quartz tube decreases. This phenomenon is explained by the intensification of ionization and excitation processes near the RF electrode, where the electric field is maximum. As a result of the increase in power, the plasma is concentrated in a more limited volume and ensures effective maintenance of the discharge with a shorter length of luminescence [15]. An increase in radiofrequency radiation power leads to an increase in the density and intensification of electron impact processes, which enhance CH_4 dissociation and increase the concentration of chemically active radicals. These processes increase losses in inelastic collisions and contribute to the spatial localization of visible plasma radiation. The power range of 11–15 W was initially selected to study discharge behavior

under relatively stable plasma conditions. In subsequent experiments, the power was increased to 20 W to study the behavior of plasma at higher excitation levels and to analyze the effect of increased plasma density on the interaction between plasma and the catalyst, which will be discussed further.

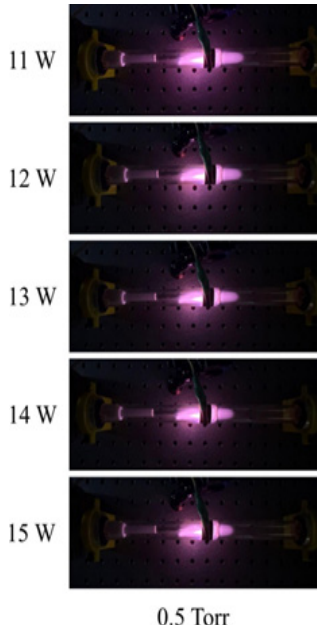

Figure 2 – Photographs of the RF-DBD discharge with the catalyst at different RF power levels, at a constant pressure of 0.5 Torr and a gas flow rate of Ar (80 sccm) + CH_4 (25 sccm)

Figure 3 shows the results of RF-DBD discharge luminescence at a constant power of 20 W and a pressure of 0.5 Torr, depending on the change in the CH_4 fraction from 10 sccm to 25 sccm in 5 sccm increments in an Ar/CH_4 gas mixture. As the CH_4 flow rate increases, the probability of electron-molecular collisions increases, and a significant part of the electron energy begins to be spent on the excitation and dissociation of CH_4. The proportion of electron energy going to the excitation of argon decreases, and as a result, the luminescence associated with Ar emission becomes less pronounced and localized [15]. The catalyst located 1.5 cm from the RF electrode has an additional effect. At higher CH_4 fractions, when the concentration of radicals and molecular products increases, plasma-catalytic processes intensify and also contribute to the spatial localization of the discharge.

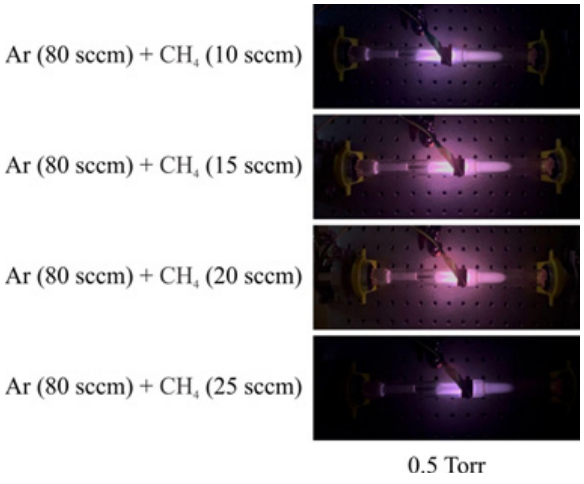

Figure 3 – Photograph of the RF-DBD discharge with the catalyst positioned 1.5 cm from the RF electrode at different gas flow rates, at a constant power of 20 W and a constant pressure of 0.5 Torr

Photos of RF-DBD discharge at a constant pressure of 0.5 Torr and a power of 20 W with the catalyst located 3.0 cm from the RF electrode are shown in Figure 4. In this case, the trend is the same as in Figure 3, but it can be seen that as the CH4 fraction increases, the effect of the catalyst on the discharge structure weakens. This is due to the increase in the distance from the RF electrode. With an increase in gas flow rate CH4=20 sccm at a power of 20 W, there is a visible absence of glow behind the catalyst, i.e., the plasma does not spread into the area beyond the nickel foam.

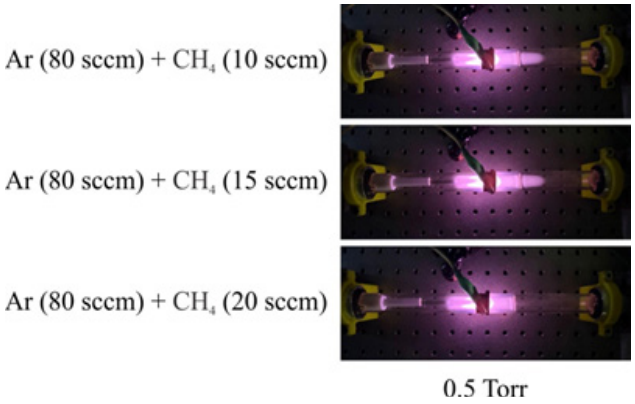

Figure 4 – Photographs of the RF-DBD discharge with the catalyst placed 3.0 cm from the RF electrode at different gas flow rates, under a constant RF power of 20 W and a constant pressure of 0.5 Torr

Spectral images of Ar/CH_4 plasma were taken with catalysts at distances of 1.5 cm and 3 cm. The spectral lines at a distance of 3 cm remain at the noise level, so the spectrum shown below was used, assuming that the catalyst was at a distance of 1.5 cm. Spectral lines of RF-DBD discharge at a pressure of 0.5 Torr and a gas flow of Ar (80 sccm) + CH_4 (25 sccm) before and after the catalyst were obtained using an optical emission spectrometer. The analysis of the spectral lines before (Figure 5 (a)) and after (Figure 5 (b)) the catalyst in the cases mentioned earlier is shown in Figure 5. The intensity of the C_2 bands (Swan bands, ~510–560 nm) before the catalyst is more intense, while after the catalyst it is noticeably reduced; the CH lines (~431 nm) also weaken after the catalyst [13, 16–19]. This suggests that active hydrocarbon radicals are consumed on the surface of nickel foam. The intensity of the atomic hydrogen peak Hα (656.3 nm) is weak, and after the catalyst, its peak remains at the noise level. The intensity of molecular hydrogen H_2 (about 600–620 nm) decreases in the zone after the catalyst but is still present [20].

The decrease in the intensity of carbon-containing bands after the catalyst indicates the effectiveness of the catalytic effect of nickel foam in plasma during CH_4 decomposition. This reflects the degree of interaction with nickel foam and is explained by the mechanism of carbon material deposition on its surface. The presence of Fulcher bands of molecular hydrogen is a key result, as it serves as direct spectral evidence of hydrogen generation. The decrease in molecular and atomic hydrogen is explained by recombination and quenching on the surface of nickel foam [20]. The authors explained the same decrease in molecular hydrogen using a nickel catalyst [21]. The authors explained this by the ability of metallic nickel to undergo hydrogenation, in which the hydrogen formed actively recombines on the surface of the catalyst.

To further confirm the catalytic effect of nickel foam, Raman spectroscopy studies were conducted after its use in Ar/CH_4 plasma.

Figure 6 shows the Raman spectroscopy spectrum of nickel foam after plasma exposure. The spectrum shows characteristic D (defect) and G (graphite) bands, whose maxima were determined to be approximately ~1375 cm^{-1} and ~1600 cm^{-1}, corresponding to amorphous carbon. The presence of these peaks indicates the formation of carbon deposits on the surface of nickel foam during the plasma-catalytic decomposition of methane [10].

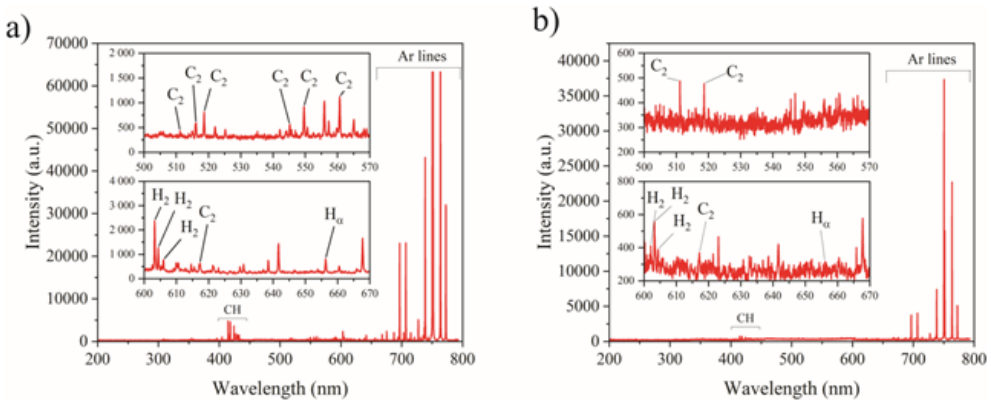

Figure 5 – Results of RF-DBD discharge spectral lines at a pressure of 0.5 Torr and a gas flow of Ar (80 sccm) + CH_4 (25 sccm): a) area before the catalyst; b) area after the catalyst

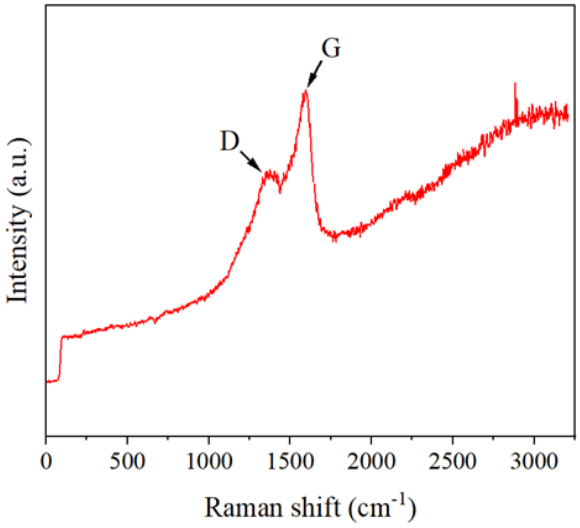

Figure 6 – Raman analysis of the catalyst (Ni foam) after the experiments

Conclusion

This study demonstrates that the parameters of RF-DBD in a low-pressure Ar/CH_4 mixture are strongly influenced by the supplied power, gas composition, and the spatial configuration of the plasma-catalytic system. In particular, the distance between the RF electrode and the nickel foam catalyst the electric field distribution and the conditions for sustaining the discharge along the reactor.

It was established that an increase in the CH_4 content is accompanied by compression of the luminous region. At a fixed power of 20 W, increasing the distance between the RF electrode and the catalyst from 1.5 cm to 3.0 cm made it impossible to sustain the plasma downstream of the nickel foam. OES revealed that at a distance of 3 cm, the spectral signal downstream of the catalyst was at the noise level. Consequently, all subsequent experiments were conducted at the optimal distance of 1.5 cm for investigating plasma-catalytic reactions. OES analysis showed a decrease in the intensity of carbon and hydrocarbon spectral lines in the plasma region after the catalyst, confirming its catalytic activity. The presence of molecular hydrogen was detected in the Ar/CH_4 plasma; however, its line intensity decreased downstream of the catalyst. This is attributed to the hydrogenation effect facilitated by the nickel surface. Furthermore, Raman spectroscopy confirmed the formation of

amorphous carbon deposits on the nickel foam, which is a significant factor influencing its catalytic properties.

The obtained results highlight the efficacy of nickel foam as an active catalytic surface for plasma-chemical reactions. A key finding is the detection of hydrogen generation via OES, underscoring the dynamic role of the catalyst in the reaction process. These findings contribute to a better understanding of plasma–catalyst interactions in RF-DBD systems and may be useful for the development of plasma-assisted CH_4 conversion technologies.

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ТӨМЕН ҚЫСЫМДА НИКЕЛЬ КӨБІГІ БАР КЕЗДЕ AR/CH_4 ҚОСПАСЫНДАҒЫ RF-DBD РАЗРЯДЫН ЗЕРТТЕУ

Аңдатпа

Бұл жұмыста никель көбігінің 0,5 Торр төмен қысымда аргон-метан қоспасындағы RF-DBD разрядының құрылымы мен қасиеттеріне әсері зерттелді. Берілген қуаттың, газ ағынының және никель көбігі мен радиожиілікті (РЖ) электрод арасындағы қашықтықтың өзгеруі бойынша эксперименттер жүргізілді. Катализатор болған жағдайда плазма қуатының артуы және метан газы ағынының өзгеруі кварц түтігіндегі плазманың жарқырау ұзындығының айтарлықтай қысқаруына әкелетіні анықталды. Катализатор мен радиожиілікті электрод арасындағы қашықтықтың ұлғаюы катализатордан кейінгі аймақта плазманы ұстап тұру мүмкіндігін төмендететіні көрсетілді. Оптикалық эмиссиялық спектрлерді талдау нәтижесінде катализатордан кейін көміртегі бар радикалдардың, атомдық және молекулалық сутектің қарқындылығының төмендеуі байқалды, бұл оның плазмалық-каталитикалық процестерге белсенді қатысатынын көрсетеді. Раман талдауы никель көбігі бетінде аморфты көміртегі шөгінділерінің түзілетінін растады. Никель көбігі разрядтың RF-DBD құрылымын өзгертіп қана қоймай, плазмадағы белсенді бөлшектердің таралуына елеулі әсер етіп, плазмалық-каталитикалық реакциялардың жүру жағдайларын өзгертетіні анықталды. Алынған нәтижелер кеуекті металл катализаторымен төмен температуралы плазманың өзара әрекеттесу механизмдерін тереңірек түсінуге мүмкіндік береді және көмірсутек газдарын конверсиялауға арналған тиімді плазмалық-каталитикалық жүйелерді әзірлеуде қолданылуы мүмкін.

Тірек сөздер: плазмалық катализ, RF-DBD разряд, оптикалық эмиссиялық спектроскопия, катализатор, никель көбігі.

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ИССЛЕДОВАНИЕ RF-DBD РАЗРЯДА В СМЕСИ AR/CH_4 В ПРИСУТСТВИИ НИКЕЛЕВОЙ ПЕНЫ ПРИ НИЗКОМ ДАВЛЕНИИ

Аннотация

В данной работе исследовано влияние никелевой пенки на структуру и свойства RF-DBD разряда в смеси аргон-метан при низком давлении 0.5 Торр. Были проведены эксперименты при варьировании подводимой мощности, потока газа и расстояния между катализатором и радиочастотным (РЧ) электродом. Показано, что увеличение мощности плазмы и изменение потока газа метана при присутствии катализатора приводит к заметному укорочению длины свечения плазмы в кварцевой трубке. Было установлено, что увеличение расстояния между катализатором и РЧ-электродом снижает возможность поддержания плазмы в области после катализатора. По итогу анализа оптических эмиссионных спектров выявлено снижение интенсивности углеродосодержащих радикалов, атомарного и молекулярного водорода после катализатора, что свидетельствует о его активном участии в плазменно-каталитических процессах. Рамановский анализ подтвердил образование отложений аморфного углерода на поверхности никелевой пенки. Установлено, что никелевая пена не только модифицирует структуру RF-DBD разряда, но и существенно влияет на распределение активных частиц в плазме, изменяя условия протекания плазменно-каталитических реакций. Полученные результаты позволяют глубже понять механизмы взаимодействия низкотемпературной плазмы с пористым металлическим катализатором и могут быть использованы при разработке эффективных плазменно-каталитических систем конверсии углеводородных газов.

Ключевые слова: плазменный катализ, RF-DBD разряд, оптическая эмиссионная спектроскопия, катализатор, никелевая пенка.