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# PLASMA CONVERSION OF $CO_2$ IN DC GLOW DISCHARGE WITH DISTRIBUTED GAS INJECTION AND PUMPING[†]

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Accumulation of carbon dioxide in the Earth's atmosphere leads to an increase in the greenhouse effect and, as a consequence, to significant climate change. Thus, the demand to develop effective technologies of carbon dioxide conversion grows year to year. Additional reason for research in this direction is the intention of Mars exploration, since 96% of the Martian atmosphere is just carbon dioxide, which can be a source of oxygen, rocket fuel, and raw materials for further chemical utilization. In the present paper, the plasma conversion of carbon dioxide have been studied in the dc glow discharge at the gas pressure of 5 Torr in a chamber with distributed gas injection and evacuation from the same side for the case of narrow interelectrode gap. The conversion coefficient and the energy efficiency of the conversion were determined using mass spectrometry of the exhaust gas mixture in dependence on $CO_2$ flow rate and the discharge current and voltage. Maximum conversion rate was up to 78% while the energy efficiency of the conversion was always less than 2%. It was found that the discharge at this pressure can operate in normal and abnormal modes and the transition between the modes corresponds just to the maximum value of the conversion coefficient for a given gas flow. It was shown that even in anomalous regime, when the cathode is completely covered by the discharge, the discharge contraction occurs in whole range of parameters studied. The anode glow and the plasma column outside the cathode layer occupy the central part of the discharge only that reduces the conversion efficiency. Optical emission spectra from the carbon dioxide plasma were measured in the range of 200-1000 nm, which allowed to make a conclusion that the Oxygen atom emission is mostly origins from the exited atoms appearing after dissociation rather than after electron impact excitation.
**Keywords:** carbon dioxide, plasma conversion, dc glow discharge
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In recent years, the processes of conversion of carbon dioxide ($CO_2$) have attracted considerable attention of researchers [1–3] due to at least two reasons. First, the accumulation of $CO_2$ in the Earth's atmosphere leads to an increase in the greenhouse effect and, as a consequence, to significant climate change. The main source of carbon dioxide emission into the atmosphere is energy enterprises that burn fossil fuels and organic materials. In order to reduce the concentration of $CO_2$ in the atmosphere, it is necessary both to reduce its emission by various sources and to develop effective methods for its utilization. Secondly, humanity plans to explore Mars and other planets and satellites of the solar system. However, 96% of the Martian atmosphere is $CO_2$ [3]. Under such conditions, it is possible to convert carbon dioxide molecules into carbon monoxide CO and oxygen $O_2$. In this case, carbon monoxide CO can be a raw material for further chemical utilization, and the $CO/O_2$ mixture has proven itself as a rocket fuel [3].

Plasma methods are among the most efficient for $CO_2$ conversion. Detailed studies were carried out with dielectric barrier discharges [4–6], gliding arc [7–9] and microwave [10–13] discharges. There are also a small number of studies on $CO_2$ conversion in nanosecond pulse discharge [14], corona [15, 16], glow [17–19], radiofrequency CCP [20, 21], capillary [22] and other types of discharges [23, 24]. Recently, a number of review papers have appeared (see for example [1, 2]), where the advantages and disadvantages of various methods of $CO_2$ conversion are analyzed in detail as well as applicability of various types of gas discharges for this process.

All plasma methods investigated to date can be divided into three different groups. Discharges of the first group (dielectric barrier, glow, microwave discharges, radiofrequency CCP, ICP [23]) allow to obtain high conversion coefficients (the ratio of the number of converted molecules to the initial number of $CO_2$ molecules entering the plasma volume) of more than 50%, but at the same time the energy efficiency of the conversion (the fraction of the discharge power spent specifically for the conversion of $CO_2$ molecules) is of the order of 1–10% and lower. The second group of discharges (gliding arcs) gives the opposite results – the energy efficiency of the conversion can exceed 50%, but the conversion rate is usually small and comparable to 10%. In the discharges of the third group (nanosecond pulse, corona, pulsed corona discharges [25]), both the conversion rate (less than 30%) and the energy efficiency of the conversion (less than 10%) are quite low. Obviously, these three groups can hardly be called effective for an economically viable production process for the conversion of carbon dioxide. Therefore, additional searches for optimal plasma technologies are needed. To do this, it is necessary to find out what types of discharges and chamber geometries will allow one to simultaneously obtain high values of both the conversion rate and the energy efficiency of the conversion.

In our recent work [19], the process of $CO_2$ conversion in DC discharge performed in the device possessing the distributed same-side gas supply and pumping for the distance between the electrodes of 60 mm was already investigated. In that work, the conversion rate reached 70%, but the energy efficiency of the conversion remained low

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and usually did not exceed 1–3%. The reason for this was revealed in [26], where it was shown using the COMSOL software that with the large gap between the electrodes (as was the case in [19]), gas molecules between entering the discharge chamber and exit into the pumping system spend longer time in the discharge chamber than is necessary for their effective conversion in plasma that leads to excessive power loss. Therefore, in this work, we investigated the $CO_2$ conversion process for lower distance between the electrodes (11 mm).

## EXPERIMENTAL

For the experiments, a discharge chamber was used, in which the $CO_2$ feeding and reaction products evacuation are performed through the same electrode (shower-like electrode, see photo in Fig. 1). This electrode had 265 holes evenly distributed over its surface. The inlet and outlet holes are located on a regular hexagonal grid with a step of 5 mm along the diagonals. This electrode was grounded to prevent parasitic discharges in the holes and in the pipes connected to it. The second, solid electrode was supplied with a positive voltage from the DC power supply; therefore, it was the anode. The electrodes were made of stainless steel.

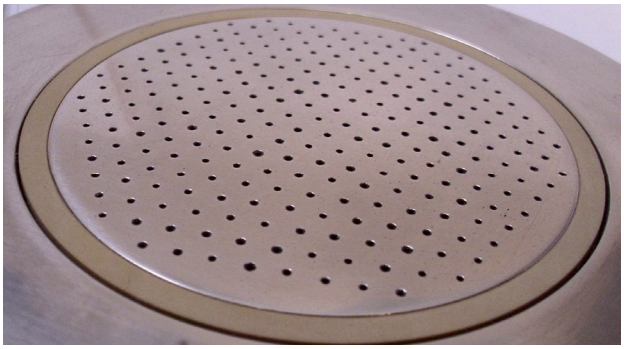

**Figure 1**. Photo of shower-like electrode

Turbomolecular and rotary vane pumps were used to evacuate the vacuum chamber. The electrodes described above were external, a section of a quartz tube with an inner diameter of 93 mm was sealed between the electrodes spaced by 11 mm gap. Note that in narrower gaps the ignition of the discharge will be difficult, since the breakdown curves are shifted to the range of high gas pressures [27, 28]. The experiments were carried out at carbon dioxide pressure of 5 Torr. To measure the gas pressure, a Baratron 627 capacitive manometer (MKS Instruments) with a maximum measured value of 10 Torr was used. The carbon dioxide flux $Q$ varied from 1 sccm to 200 sccm using a mass-flow controller.

The $CO_2$ conversion rate can be estimated using mass spectrometry of the exhaust gas. The gas mixture leaving the discharge chamber consists mainly of $CO_2$, CO, O and $O_2$. Using a thin capillary with inner diameter of 1 mm, the analyzed portion of gas was taken from the pumping system near the outlet of the discharge chamber and fed through a valve into the ROMS-4 mass spectrometer pumped by separate ion pump. Note that oxygen atoms recombine on the walls of the capillary with the formation of $O_2$ molecules. The ion peak in the mass spectrum corresponding to the 16th mass ($O^+$) origins from $O_2$ molecules dissociation in the mass spectrometer ionizer. Therefore, we analyzed the peaks of M = 28 ($CO^+$), M = 32 ($O_2^+$) and M = 44 ($CO_2^+$) mass. Before each experiment, the mass spectrum of the residual gas in the mass spectrometer was measured and then subtracted from the obtained mass spectra of the analyzed gas mixture.

The optical emission of the discharge was measured by optical emission spectroscopy. The optical fiber was in a fixed position 5 mm from the grounded cathode electrode (almost in the center of the gap between the electrodes) and supplied the collected discharge radiation to the Horiba iHR-320 optical spectrometer. This spectrometer has a diffraction grating of 1800 lines/mm allowing spectrum measurement in wide wavelength range (200–1000 nm), with high resolution (better then 1 Å).

Note that the following three parameters are used to characterize the process of carbon dioxide conversion: specific energy input (SEI), absolute conversion coefficient $\chi$ and energy efficiency of the process $\eta$. Specific energy input is the ratio of the power supplied to the discharge to the value of the gas flow:

$$SEI\left[J\cdot cm^{-3}\right]=\frac{P\left[kW\right]}{\frac{dm}{dt}\left[L\cdot \min^{-1}\right]}\cdot 60\left[s\cdot \min^{-1}\right]\times$$
$$\times\frac{6.24\cdot 10^{21}\left[eV\cdot kJ^{-1}\right]\cdot 24.5\left[L\cdot mol^{-1}\right]}{6.022\cdot 10^{23}\left[mol^{-1}\right]},$$

where $P$ is the power, $dm/dt$ is the gas flow rate. The absolute conversion rate is the ratio of the number of $CO_2$ molecules that have been converted as the gas passes through the plasma volume to their initial number:

$$\chi = \frac{N_{in} - N_{out}}{N_{in}},$$

where $N_{in}$ and $N_{out}$ are $CO_2$ fluxes entering and pumping out of the chamber, respectively. Energy efficiency compares the energy consumption of a given conversion technique with the standard enthalpy of the process:

$$\eta = \chi \cdot \frac{\Delta H}{SEI},$$

where $\Delta H = 2.93$ eV per molecule.

Thus, SEI, conversion coefficient χ and energy efficiency η can be determined if the concentration of $CO_2$ molecules without plasma and with a burning discharge were measured using a mass spectrometer, while simultaneously registering the power $P$ deposited into the plasma and gas flow $Q$ fed into the chamber. This technique is described in detail in [23, 24].

## EXPERIMENTAL RESULTS

Let us consider our results obtained for a carbon dioxide pressure of 5 Torr. This pressure is interesting because the average atmospheric pressure of Mars is close to this value, therefore, a significant part of the experiments of other authors on the conversion of $CO_2$ were carried out precisely for this pressure.

Figure 2 shows a photograph of the discharge with current of 100 mA for the considered $CO_2$ pressure. At a lower current the normal mode is observed where the discharge covers only a part of the cathode surface.

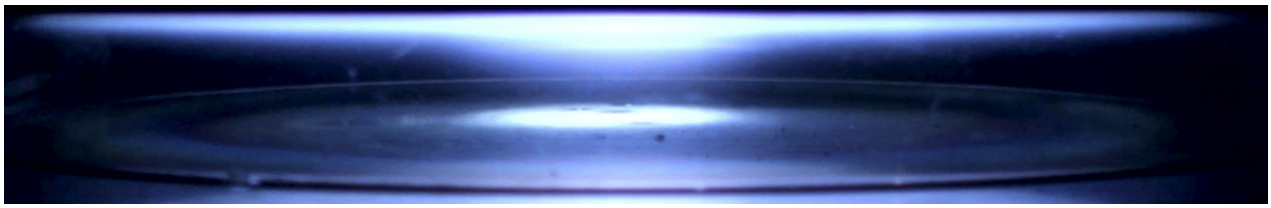

**Figure 2.** Photo of the discharge at $CO_2$ pressure of 5 Torr, gas flow rate of 2 sccm, and discharge current of 100 mA.

In this case, an increase in the current (until the moment of complete coverage of the entire cathode by the discharge) occurs at a constant or even decreasing voltage across the electrodes [29]. Figure 3 shows that the current-voltage characteristics of the discharge have two branches: falling (the current decreases with increasing voltage) and growing (current and voltage increase simultaneously).

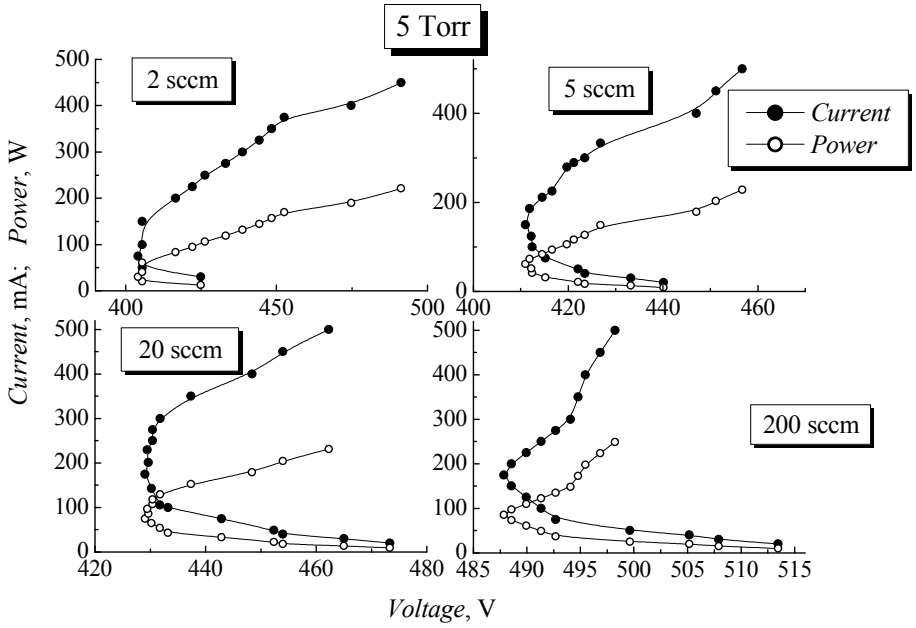


**Figure 3.** Dependences of the current and the power supplied to the discharge on the applied voltage at various gas flow rates (2 sccm, 5 sccm, 20 sccm and 200 sccm).

The falling branch belongs to the normal mode. The growing branch describes an anomalous regime in which the cathode surface is already completely covered by the discharge, and to further increase the current, it is necessary to increase the voltage across the cathode layer (and over the entire discharge gap) to enhance the ionization production of

charged particles. The values of the power supplied to the discharge presented in Fig. 3 will be used below to determine the SEI and the energy efficiency η of the process.

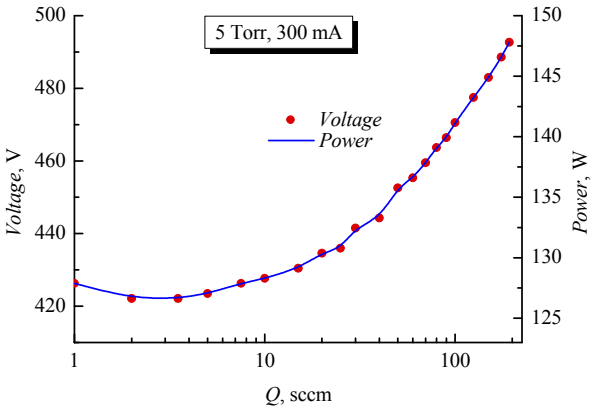


**Figure 4.** Dependence of the voltage across the electrodes and the power applied to the discharge on the gas flow $Q$ at the pressure of 5 Torr and the current of 300 mA

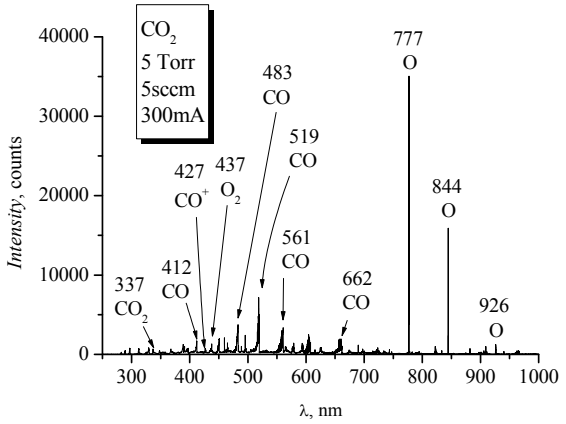


**Figure 5.** Optical emission spectrum measured at carbon dioxide pressure of 5 Torr, flow rate of 5 sccm, and discharge current of 300 mA.

Note that even when the discharge completely covers the cathode surface the glow occupies only a part of the anode area. If the distance between the electrodes is increased, then, starting from the anode, with a sufficiently large gap, a contracted positive column is formed, which occupies only a part of the cross section of the discharge tube. The photograph of the discharge shown in Fig. 2 shows that the cathode layer (which is practically invisible) and the negative glow jointly extend only 2–3 mm from the cathode surface. Deceleration of the bulk of fast electrons (born and accelerated in the strong electric field of the cathode layer) occurs just in the negative glow. The negative glow transforms into the dark Faraday space and then, in the region of the plasma column, into the anode glow. However, outside this column, a significant part of the discharge volume is occupied by decaying plasma with low density of charged particles. Therefore, as will be shown below, at a carbon dioxide pressure of 5 Torr, the energy efficiency of the conversion is low.

Figure 4 shows that with an increase in the carbon dioxide flux $Q$ both the voltage between the electrodes and the power applied to the discharge first decrease, reach a minimum at a flux of about 2.5–3 sccm, and then increase. The discharge current during this experiment was kept constant.

The optical emission spectrum of the discharge (see Fig. 5) consists of lines of atomic oxygen O (777 nm, 844 nm and 926 nm), as well as a large number of bands of CO molecules (they mainly relate to the Angstrom system, the $B\ ^1\Sigma \rightarrow A\ ^1\Pi$ transition from the second to the first state of electronic excitation of the molecule).

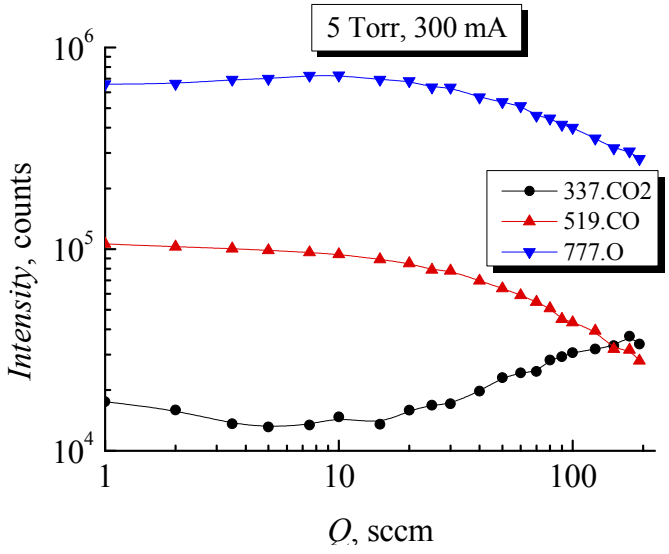


**Figure 6.** Dependences of the intensities of emission lines for $CO_2$ (337 nm), CO (519 nm) and O (777 nm) on the gas flow rate at pressure of 5 Torr and discharge current of 300 mA.

In addition, there are weak lines of $O_2$ (Schumann-Runge system, $B\ ^3\Sigma \rightarrow X\ ^3\Sigma$), $CO^+$ ("comet tail" system, $A\ ^2\Pi \rightarrow\ ^2\Sigma$ transition from the first excited electronic state to the ground state). The glow of $CO_2$ molecules is

represented by a weak line with a wavelength of 337 nm. Note that the luminescence of atomic oxygen can be caused both by the dissociation process itself (in this case, the $CO_2$ molecule decays into CO in the ground state and into an excited O atom [1, 2]), and by the excitation of oxygen atoms by subsequent electron impacts. That is why the intensities of the lines of atomic oxygen are so high in comparison with the lines of CO molecules. Moreover, the intensities of the CO, $CO_2$ and $O_2$ lines (which are excited only by electron impact) are usually tens of times lower than the intensities of the atomic oxygen lines. Thus, we suppose that the glow of oxygen atoms at wavelengths of 777 nm and 844 nm is almost completely associated with the process of dissociation of $CO_2$ molecules.

The difference in the mechanisms of excitation of CO molecules and O atoms is clearly seen in Fig. 6, which shows the dependences of the intensities of the emission lines of $CO_2$, CO and O on the gas flow rate.

It follows from the figure that the emission intensity of carbon dioxide molecules first decreases with the gas flow rate increase, reaches a minimum, and then increases. The opposite behavior is shown by the intensity of the lines of oxygen atoms, first increasing with the flow rate increase, then reaching a maximum, and finally decreasing. This behavior of the $CO_2$ and O lines can be explained by the assumption that at low gas flow rates ($Q \sim 1$ sccm) carbon dioxide molecules have time not only to be converted into CO and O, but also to partially recombine back into $CO_2$ molecules. With the flow rate increase, the residence time of molecules in the discharge chamber decreases, the process of CO and O recombination in $CO_2$ plays a lesser role, thus, the $CO_2$ concentration decreases, and the concentration of oxygen atoms increases. At higher flows ($Q > 10$ sccm), the gas is removed from the discharge without having time to be converted. However, the intensity of the CO line decreases monotonically with increasing gas flow, without reaching any extrema. Therefore, a clear correlation between the behavior of the $CO_2$ and O intensities is associated with the fact that the loss of $CO_2$ molecules during dissociation is accompanied by the appearance of excited O atoms. But CO molecules emit light only after they experience inelastic collisions with electrons after the conversion. Consequently, the intensity of their luminescence decreases with an increase in the gas flow due to the intensification of their removal from the discharge chamber.

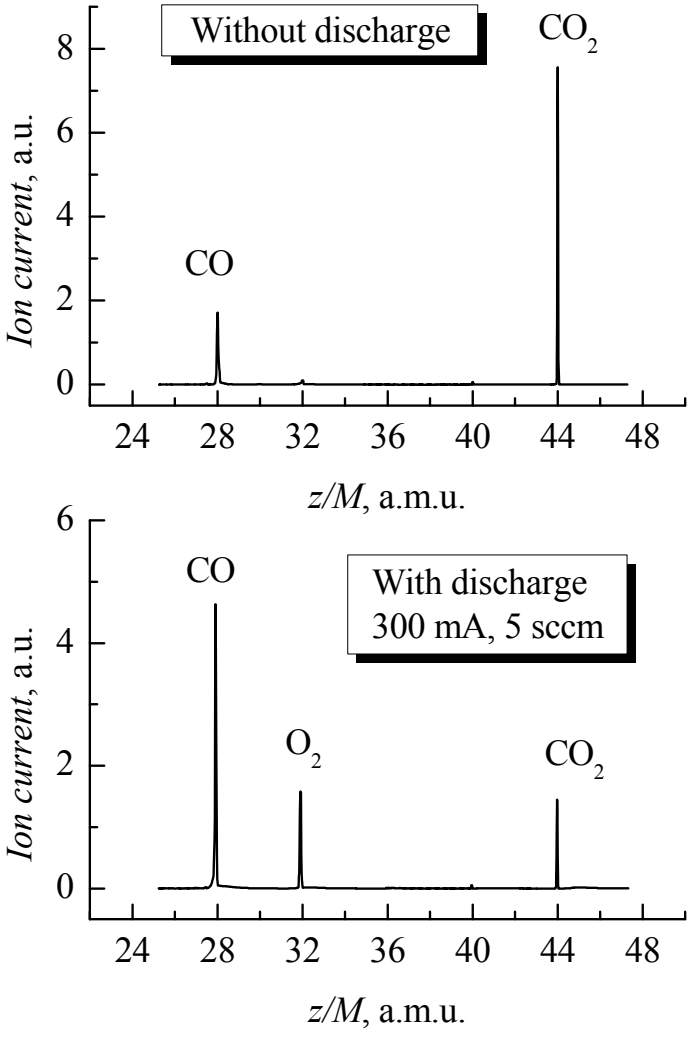


**Figure 7.** Mass spectra of the gas mixture leaving the discharge on and off. The gas flow rate is 5 sccm. The discharge current is 300 mA.

Now let us consider the mass spectra of the exhaust gas mixture (see Fig. 7). The mass spectrum of the gas leaving the chamber without discharge should only consist of $CO_2^+$ (M = 44). The $CO^+$ peak (M = 28) and a weak $O_2^+$ peak (M = 32) are observed, which are formed inside the mass spectrometer as a result of $CO_2$ dissociation and subsequent ionization of its products. The appearance of plasma in the chamber leads to a significant decrease in the $CO_2^+$ peak and a simultaneous increase of $CO^+$ and $O_2^+$ peaks.

From the measured mass spectra, the intensities of the $CO_2^+$, $CO^+$ and $O_2^+$ peaks were determined. Fig. 8 shows the dependences of the intensities of these peaks on the gas flow. It can be seen from the figure that the lowest values of the $CO_2^+$ peak are observed at low gas flows, and with increasing $Q$ the $CO_2^+$ peak grows. This indicates that an increase in gas flow leads to the fact that more and more carbon dioxide molecules are removed from the discharge chamber, without having time and enough power to convert into CO and O.

Using the measured mass spectra and current-voltage characteristics of the glow discharge, the values of the conversion coefficient $\chi$ and the energy efficiency of the conversion $\eta$ were determined, the dependences of which on the gas flow rate are shown in Fig. 9. It can be seen from the figure that at low gas flows the conversion coefficient reaches 78%, but with an increase in $Q$ the conversion coefficient monotonically decreases down to 23% at

$Q$ = 100 sccm due to a decrease in the residence time of $CO_2$ molecules in the discharge. The energy efficiency of conversion η at low flow rates is quite low and amounts to only 0.07%, but at high flows it reaches 1.6%.

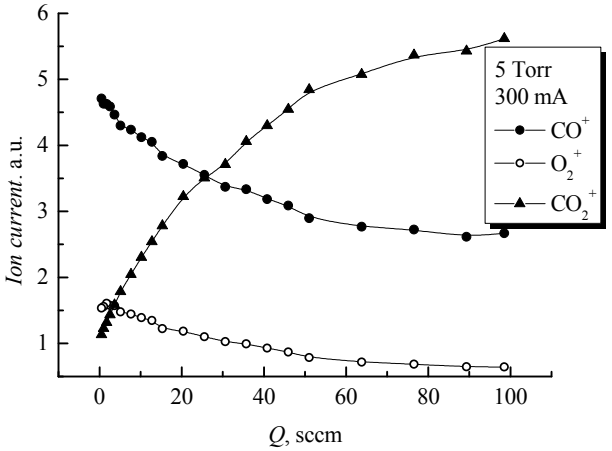


**Figure 8.** Dependences of the peak intensities in the mass spectra of the gas mixture leaving the discharge chamber into the pumping system on the gas flow at the gas pressure of 5 Torr and discharge current of 300 mA.

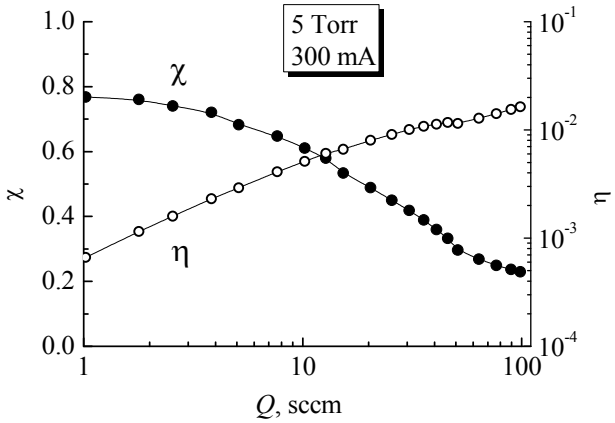


**Figure 9.** Dependences of the conversion coefficient χ and the energy efficiency of the conversion η on the gas flow at pressure of 5 Torr and discharge current of 300 mA.

Since the specific energy input SEI is inversely proportional to the gas flow, the dependences of χ and η on the gas flow rate shown in Fig. 9 are mirrored when plotting them as a function of SEI (see Fig. 10). That is, the low $Q$ range corresponds to high SEI values. Conversely, at high gas flows the SEI becomes small. It should be kept in mind that SEI is proportional to the power input into the discharge, which depends on the gas flow (see Fig. 4).

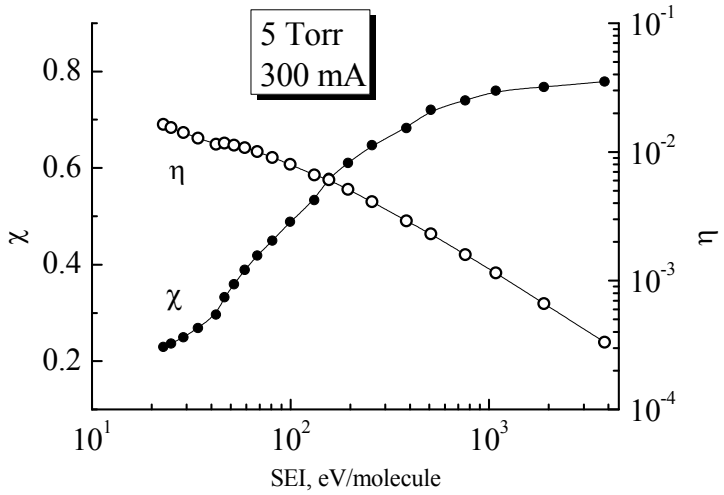


**Figure 10.** Dependences of the conversion coefficient χ and the energy efficiency of conversion η on SEI at the pressure of 5 Torr and the discharge current of 300 mA.

A convenient tool for comparison of carbon dioxide conversion processes under various conditions (types of discharges, ranges of gas pressure, current, gas flow, etc.) is the dependence of η on χ. Such dependence at fixed pressure and discharge current, but different gas flow rates, is shown in Fig. 11. From the economical point of view, the most optimal case is when both η and χ are close to 100%. However, at present, such a technology for the conversion of carbon dioxide has not yet been developed, as we mentioned in the Introduction. It follows from Figure 11 that higher values of the energy efficiency η are observed at low values of the conversion coefficient χ. An increase in χ is accompanied by a rapid decrease in energy efficiency. Under the conditions of our experiments, it was possible to achieve the maximum conversion rate χ = 78%, but at the same time the energy efficiency of the conversion is approximately equal to 0.07%.

A similar dependence of η on χ at the 5 Torr pressure with various flow rates is shown in Fig. 12. To obtain these dependences, the current varied from the minimum value at which it was possible to maintain a stable discharge burning (several milliamperes) to 500 mA. Recall that at low currents, the glow discharge burns in the normal mode, covering only a part of the cathode surface. The complete filling of the cathode with a discharge is observed at approximately a current of 100–200 mA (which depends on the gas flow). From Fig. 12 it can be seen that for a flow of $Q$ = 1 sccm, the dependence of η on χ has two branches: an upper (corresponding to low currents) and a lower (high-current branch).

The transition from the upper to the lower branch occurs at the maximum value of the conversion coefficient χ for a given gas flow. The more the gas flow was, the less pronounced the lower branch was. In this case, the use of higher gas flows led to an increase in the energy efficiency of the conversion η. At a flow rate of $Q$ = 25 sccm, it was possible to simultaneously achieve the values χ > 50% and η ≈ 1 %; moreover, the value of the energy efficiency η changed little over a wide range of values of the discharge current. Note that the presented results on the conversion efficiency are generally close to the same results of the paper [19] with wider discharge gap.

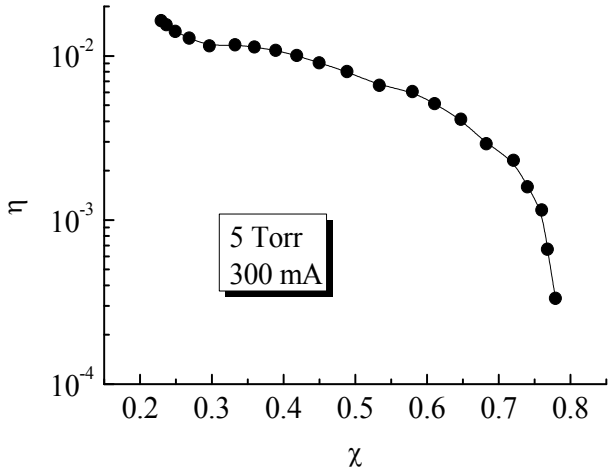


**Figure 11**. Relation between the energy efficiency of conversion η and the conversion coefficient χ at a pressure of 5 Torr and a discharge current of 300 mA.

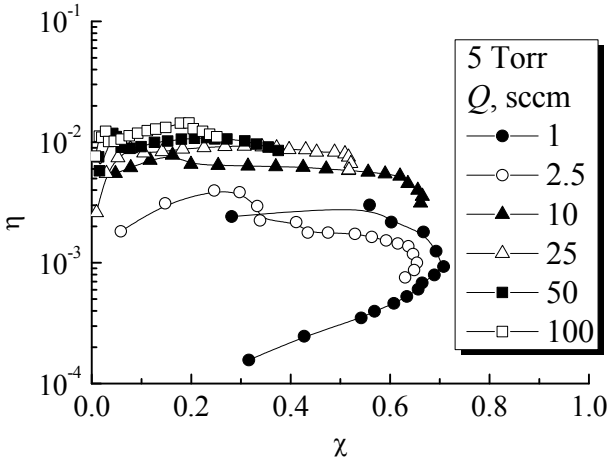


**Figure 12.** Dependence of the energy efficiency of conversion η on the conversion coefficient χ at the 5 Torr pressure with various gas flow rates

## CONCLUSION

In the present research, we have studied the conversion of carbon dioxide in the dc glow discharge at the gas pressure of 5 Torr in a chamber with distributed gas injection and evacuation from the same side for the case of narrow interelectrode gap. The conversion coefficient χ and the energy efficiency of the conversion η were determined using mass spectrometry of the exhaust gas mixture in dependence on $CO_2$ flow rate and the discharge current and voltage. Under the conditions of our experiments, it was possible to achieve the maximum conversion rate χ = 78%, but the energy efficiency of the conversion was always less then 2%. Optical emission spectra from the carbon dioxide plasma were measured in the range of 200-1000 nm, which allowed to make a conclusion that the oxygen atom emission is mostly origins from the excited atoms appearing after dissociation rather than after electron impact excitation.

It is shown that at the studied pressure the discharge can operate in two different modes. At low discharge current the normal mode reveals where the discharge covers only a part of the cathode. At higher currents the anomalous regime appears, in which the cathode surface is already completely covered by the discharge, and to further increase the current, it is necessary to increase the discharge voltage. We have found that the transition between the modes occurs just at the maximum value of the conversion coefficient χ for a given gas flow. The use of higher gas flows led to an increase in the energy efficiency of the conversion η. At a flow rate of Q = 25 sccm, it was possible to simultaneously achieve the values χ > 50% and η ≈ 1 %.

It was found that even in anomalous regime, when the cathode is completely covered by the discharge, the discharge contraction occurs in whole range of parameters studied. The plasma column outside the cathode layer and the anode glow occupy the central part of the discharge only. Outside this column, in a significant part of the discharge volume only low density decaying plasma is present that reduces the energy efficiency of the conversion.

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## ПЛАЗМОВА КОНВЕРСІЯ $CO_2$ У ТЛІЮЧОМУ РОЗРЯДІ ПОСТІЙНОГО СТРУМУ З РОЗПОДІЛЕНИМ НАПУСКОМ ТА ВІДКАЧУВАННЯМ ГАЗУ

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Накопичення вуглекислого газу в атмосфері Землі призводить до посилення парникового ефекту і, як наслідок, до значної зміни клімату. Таким чином, попит на розробку ефективних технологій конверсії вуглекислого газу з кожним роком зростає. Додатковим приводом для досліджень у цьому напрямку є намір дослідження Марса, оскільки 96% марсіанської атмосфери — це вуглекислий газ, який може бути джерелом кисню, ракетного палива та сировини для подальшої хімічної утилізації. У цій роботі досліджено плазмова конверсія вуглекислого газу в тліючому розряді постійного струму при тиску газу 5 Торр в камері з розподіленим напуском та відкачуванням газу з одного боку для випадку вузького міжелектродного проміжку. Коефіцієнт конверсії та її енергоефективність визначали за допомогою мас-спектрометрії суміші вихлопних газів залежно від потоку $CO_2$ та струму та напруги розряду. Максимальний коефіцієнт конверсії становив до 78%, тоді як енергоефективність конверсії завжди була менша за 2%. Встановлено, що розряд при цьому тиску може існувати в нормальному і аномальному режимах, а перехід між режимами відповідає якраз максимальному значенню коефіцієнта конверсії для даного потоку газу. Показано, що навіть в аномальному режимі, коли катод повністю покритий розрядом, контракція розряду відбувається в усьому діапазоні досліджуваних параметрів. Анодне світіння і стовп плазми поза катодним шаром займають лише центральну частину розряду, що знижує ефективність конверсії. Виміряно спектри оптичного випромінювання з плазми вуглекислого газу в діапазоні 200-1000 нм, що дозволило зробити висновок, що випромінювання атома Оксигену здебільшого відбувається від збуджених атомів, які виникають після дисоціації, а не після збудження електронним ударом.

**Ключові слова:** вуглекислий газ, плазмова конверсія, тліючий розряд постійного струму