

PLASMA CHEMISTRY OF SO_x IN A PULSED ELECTRIC DISCHARGE AT ATMOSPHERE PRESSURE

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The theoretical and experimental investigations of gases SO_2 and SO_3 dissociation by using the pulsed spark discharge at atmosphere pressure are represented. The production of SO_x (SO_2 , SO_3) was performed in a special device by means of electric arc discharge at the sulphur burning in gases mixture of 80% O_2 and 20% Ar . The efficient decay of SO_x radicals in the pulsed spark discharge jumping between a plate and a set of eagles has been demonstrated. It has been shown that the multispark discharge decomposes SO_x radicals by more than 20% during tens minutes with a simultaneous release of decomposition products from the gas phase of substance.

1. INTRODUCTION

The pulsed spark discharge at atmosphere pressure has been considered as a suitable for plasma chemistry [1] in particular for dissociation processes of SO_x . The "jumping" spark discharge between a set of eagles and a plate was used for the treatment of a considerable gas volume. The parameters of the discharge are the followings: voltage is 13 kV, pulsed current is 80 A, time duration is 0.8 μ sec, frequency repetition 1.5 kHz. The theoretical and experimental investigations have been performed to dissociate the gases SO_2 and SO_3 . The production of SO_x was performed in a special device by the sulphur burning in gases mixture of 80% O_2 and 20% Ar .

Two methodics have been elaborated and used for the determination of the reaction products by means of (1) amplitudes changing at mass spectrographs before and after discharge and (2) pressure and temperature changing of gases mixture in the process of discharge. The changes of pressure $\Delta P/P=22.4\%$ and temperature $\Delta T/T=3.5\%$ were observed. So from pressure changing we can determine SO_x concentration decrease (i.e. $\Delta n/n \approx \Delta P/P$), that is coincident with mass-spectra data. The value 22.7% of SO_x decrease was evaluated from the mass-spectra of initial mixture and treated one by the pulsed electric discharge during one hour. Simultaneously the crystal deposition of sulphur was observed. Performed investigations allow supposing that the optimization of SO_2 dissociation process in such a discharge can be enhanced by the removal of the decay products.

2. EXPERIMENTAL SETUP

Sulphur burning

Experiments on SO_x production were conducted in the plasma-chemical reactor, the detailed description of which is represented in Fig. 1.

The formation of SO_x products (SO_2 , SO_3) took place in the hermetically sealed glass chamber CV with sulfur burning in oxygen atmosphere (80% oxygen and 20% argon).

The chamber comprised a ceramic crucible with sulfur (6), the electrodes for burning sulfur, and the

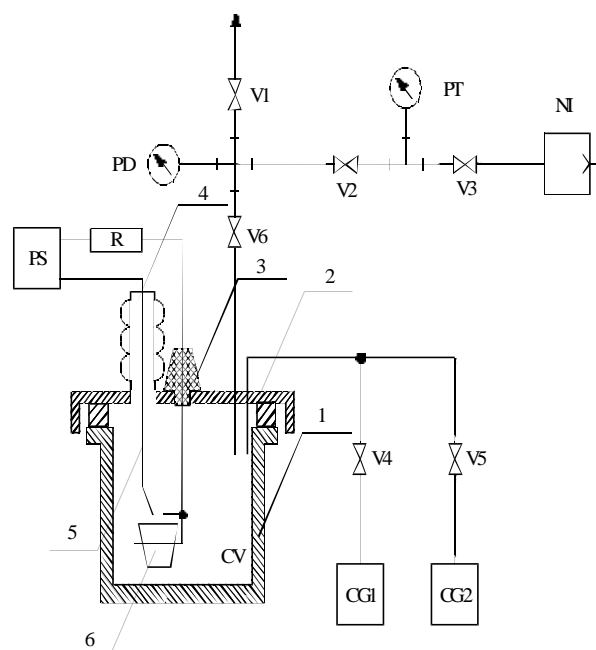
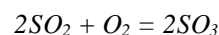


Fig. 1. Schematic of the plasma-chemical reactor: 1 - chamber body, 2 - chamber lid, 3 - insulator, 4 - bellows, 5 - movable electrode, 6 - crucible with sulphur, CV - chamber for gas mixture preparation, CG1 - chamber with oxygen, CG2 - chamber with argon, NI - mechanical vacuum pump, PD - deformation vacuum gauge, PT - thermal vacuum gauge, V1 ... V6 straightway valves, PS - power supply (voltage 600 V, peak current 0.3 A), R - limiting resistor.

metal pipes, to which the reactor and the chambers with the spent gases CG1 and CG2 were connected by the use of vacuum lines. The CV chamber was pumped down by means of a mechanical vacuum pump NI. Sulfur (3-g dosage) was burnt by means of an arc discharge. In this case nearly 100% burning of sulfur occurred. The process of sulfur burning lasted several minutes, and the chamber was filled with a misty vapor - "fog". Evidently, the following reaction takes place



It should be noted that the process of sulfur burning is accompanied by an intense mixing of gas

components being present. By the moment this process is finished, the “fog” density is so high that the electrodes, the crucible and the metal pipes in the chamber can no longer be distinguished. The transparency of gas environment is restored with time. 5 or 6 minutes after the sulfur burning is completed, the product SO_2 together with argon and oxygen remaining go, with opening the valve $V1$, to the reactor chamber. Since the volumes of both the chamber CV and the reactor are the same, the reactor is filled with the gas mixture to a pressure $P \sim 0.5$ atm. For the time of discharge action, the chamber CV is disconnected with the reactor chamber. A low-inertia thermometer was placed into the reactor to measure the average temperature of gas.

SOx radicals dissociation

Experiments on decomposition of SO_x radicals in the discharge were carried out using the facility, the schematic of which, as well as electric and measuring circuits are shown in Fig. 2.

The cylindrical kovar glass vessel (3), whose ends are soldered with the flanges, made from kovar (2, 6), simultaneously serves as a gas-discharge chamber and the chemical reactor.

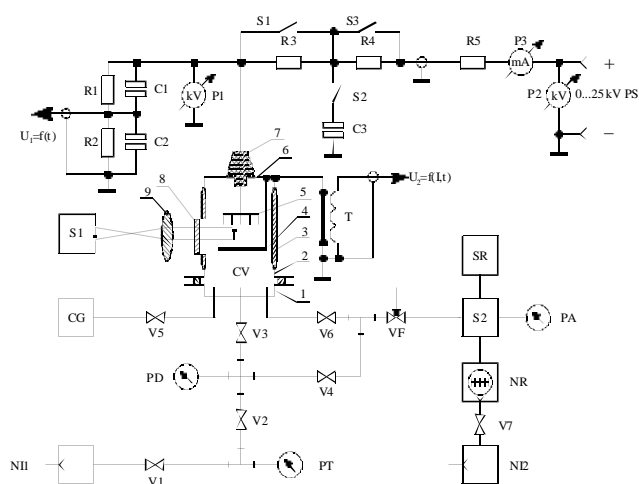


Fig. 2. Plasma chemical reactor: CV - reactor chamber, CG - working gas chamber, NII - vacuum mechanical pump NVZ-20, $NII2$ - vacuum mechanical pump 2 NVR-5DM; NR - vacuum turbomolecular pump VMN-500; $S1$ - spectrograph DFS-452, $S2$ - mass-spectrometer MX7301, SR - two-coordinate self-recorder N-306, PD - deformation vacuum gauge; PA - ionization vacuum gauge, PT - thermal vacuum gauge; $V1 \dots V7$ - straightway valves, VF - metering valve, $R1 \dots R5$ - resistors, $C1 \dots C3$ - capacitors, $P1, P2$ - kilovoltmeters C96; T - transformer (Rogowsky belt). 1 - reactor bottom, 2 - lower flange of the reactor chamber, 3 - glass case of the chamber, 4 - lower electrode, 5 - upper electrode, 6 - upper flange of the chamber, 7 - insulator, 8 - quartz window, 9 - quartz lens.

The vessel tightness was provided by vacuum rubber seals. The vessel was evacuated with the use of a forevacuum pump NVZ-20 (NII). In the wall of the vessel (at its midheight) there was a hole, $1.6 \cdot 10^{-2}$ m in diameter, to which the quartz glass window was bonded from the outside to watch the discharge radiation in visible and ultraviolet regions of wavelengths. Along the axis of the vessel two insulated electrodes (4, 5) were placed. Their leads were passed through the flange (6) and connected to the power supply. The voltage polarity could be changed from the outside of the reactor volume by switching the appropriate cables. The working volume of the reactor was $3.5 \cdot 10^{-3} \text{ m}^3$ (cylinder's diameter was 0.15 m). The electrodes were manufactured from different materials and, depending on the purposes of experiment (discharge type), were variously shaped. Generally, it was the upper electrode that was replaced, the lower electrode virtually remained the same. The lower electrode was made from aluminum; it had a shape of a mushroom with a plane cap ($3.8 \cdot 10^{-2}$ m in diameter) and rounded borders.

The other electrode, movable along the axis, was a stainless steel rod, $8 \cdot 10^{-3}$ m in diameter, with a conical end. This electrode was used to ignite the arc discharge. In another version, it was a many-needle electrode, made from the sheet material, with its working surface facing the lower electrode. Or, still in another version, it was a comb including seven copper wires ($5 \cdot 10^{-4}$ m in diameter, each being $5 \cdot 10^{-3}$ m in length), that were soldered into the copper plate at an equal spacing of $5 \cdot 10^{-3}$ m. The length of the comb itself corresponded to the diameter of the plane part of the lower electrode.

The interelectrode spacing could be varied owing to the translational motion of the upper electrode in the vacuum seal without deteriorating the hermeticity of the reactor chamber.

The electrodes of the discharge chamber CV are power supplied via ballast resistors $R3, R4, R5$ from the controlled dc source PS having a maximum power

$W = 4 \text{ kW}$ ($U_0 = 0$ to 30 kV, $I_{\max} = 150 \text{ mA}$). The

discharge chamber design provided the conditions for igniting the discharge of three types: corona and arc discharges with a constant current, and the spark discharge at a pulsed mode of operation. The types of the discharge could be varied by using the switches $S1, S2$ and $S3$ according to the scheme

Discharge	S1	S2	S3
Corona	Off	Off	Off
Arc	On	Off	On
Spark	On	On	Off

For any type of discharge, the breakdown voltage of the discharge gap of the chamber CV is proportional to the interelectrode spacing and is inversely proportional to the gas pressure in the discharge gap.

The corona-discharge mode of operation is characterized by a high voltage across the discharge gap $U = U_0$ and a low current $I \leq 1 \text{ mA}$. At the arc-

discharge mode of operation a considerable current $I \geq 15$ mA sets in at a comparatively low voltage $U = (0.1 \text{ to } 0.3) U_0$. The power source PS voltage was controlled with a kilovoltmeter $P2$, and the voltage across the discharge gap and the current values were controlled by means of $P1$ and $P3$, respectively.

The spark-discharge mode of operation was realized in the case when relaxation oscillations appeared in the electrodes circuit and their power supply. These oscillations are characterized by sharp changes in the rate of increase and decrease in the voltage and current. They (oscillations) approach a periodic sequence of pulses. The spectrum of relaxation oscillations is very wide. The relaxation oscillation generators operate at the conditions when during each period of oscillations the parameters of the circuit drastically change as a result of the occurrence of processes in it. The relaxation generator includes resistors $R_S = R_4 + R_5$, a capacitor C_3 and a discharger (interelectrode spacing of the discharge chamber CV) connected in parallel to C_3 . In this case, the breakdown potential of the discharger U_{br} should be lower than the power supply voltage U_0 . The total resistance of the resistors $R_S = R_4 + R_5$ has the value, at which the independent discharge is impossible, though the voltage U_0 value is sufficiently high. The possibility of this situation is connected with the fact that for the independent discharge in the gas-discharge device the current through it and the voltage across it must be higher than their minimum values: $I \geq I_{min}$, $U \geq U_{min}$.

The independent discharge becomes impossible if

$$R_S > \frac{U_0 - U_{min}}{I_{min}}. \quad (1)$$

For most gas-filled dischargers the condition (1) is fulfilled at $R_S > (1 \text{ to } 2) \text{ M}$. With the fulfillment of condition (1) the process reaches the steady state in the circuit (see Fig. 3a). It involves a sequence of stages as to the slow charging of the capacitor C_3 through the charge resistor R_S up to the breakdown voltage of the discharger for the time

$$t' = R_S C_3 \ln \frac{U_0 - U_d}{U_0 - U_{br}}, \quad (2)$$

(where U_d is the discharge decay potential ($U_d \approx 1$ to 2 kV), C_3 is the capacitor C_3 capacitance), and also in relation to a quick discharging through the internal resistance of the discharger for the time

$$t'' = r C_3 \ln \frac{U_{br}}{U_d}. \quad (3)$$

In the mode of operation of constant oscillations, $r \ll R_S$ at the instants of spark breakdown.

The period of relaxation oscillations is given by

$$t_0 = t' + t'' = R_S C_3 \ln \frac{U_0 - U_d}{U_0 - U_{br}} + r C_3 \ln \frac{U_{br}}{U_d}. \quad (4)$$

The charge current is

$$I_{ch} = \frac{U_0 - U_d}{R_S} \exp\left(-\frac{t}{R_S C_3}\right), \quad (5)$$

and the discharge current is calculated by

$$I_{dis} = -\frac{U_{br}}{r} \exp\left(-\frac{t}{r C_3}\right). \quad (6)$$

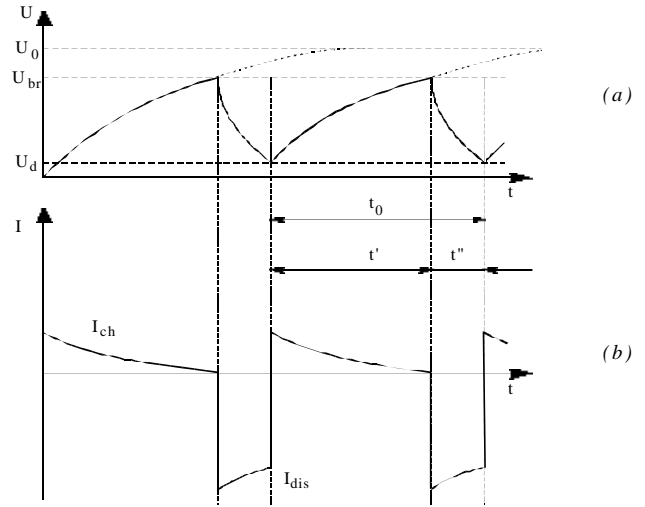


Fig. 3. Oscillograms of current (a) and voltage (b) in the spark-discharge mode.

The energy stored in the capacitor C_3 is given by

$$W = \frac{C_3 U_{br}^2}{2}. \quad (7)$$

The current variation with time in the discharger electrode circuit has a discrete character (see Fig. 3b).

As is seen from equation (4), the period of relaxation oscillations can be controlled mainly by varying the charge resistor R_S and the capacitor C_3 capacitance, and, to a less extent, by varying the voltages U_0 , U_{br} , U_d . If at a given electrode shape and a fixed gas pressure in the discharge chamber, the voltage U_{br} is proportional to the interelectrode distance and can vary in wide ranges, then the discharge decay voltage U_d remains practically uncontrolled ($U_d = Const$), because it depends on the composition of gas, its type, electrode shape, etc. So, at a fixed interelectrode spacing ($U_d = Const$), the oscillation period can be varied by varying the resistance value of R_S , the C_3 capacitance and the power supply voltage U_0 .

It should be noted that under real conditions, even at fixed R_S , C_3 and U_0 values, it appears impossible to obtain fully periodic oscillations because of a stochastic variation in the properties of the discharge gap, that results in the change of U_{br} and U_d , and, according to eqs. (2) and (3), in the variations of t' and t'' .

From relationship (6) it follows that the discharge current value is proportional to the breakdown voltage U_{br} value, while the pulse length of the discharge

current is proportional to the C_3 capacitance. It is seen from relations (6) and (7) that for the increase of pulsed energy input in the discharge the C_3 capacitance and/or the voltage U_0 should be increased. However, according to (4), as these parameter values are increased, the oscillation period also increases, and the average energy input in the discharge remains unchanged. Therefore, to increase the average energy input, the resistance of the charge resistor R_S should be reduced simultaneously with an increase in the C_3 capacitance or in the interelectrode spacing of the discharger (increase in U_d). In this case, according to (5), discharge current the capacitor C_3 will also increase to enhance the power taken away from the power source.

In the spark-discharge mode of operation, the power supply voltage U_0 is controlled by the kilovoltmeter P_2 . The breakdown voltage U_{br} , the discharge decay voltage U_d , the oscillation period are controlled and measured using the oscillograph with a memory, the voltage across which is taken at a frequency of the compensated voltage divider $R_1-R_2-C_1-C_2$ (the division coefficient $k = 10^4$). The discharge current amplitude is measured by the oscillograph with memory, the voltage to which is supplied from the calibrated current transformer T (Rogowsky belt).

Thanks to a specially made support, the plasma-chemical reactor was fixed on a rack of the diffraction spectrograph DFS-452. This fixture gave the possibility of moving the reactor upwards and downwards, to make the rotation in the horizontal plane, and also to shorten or to extend the distance from the diffraction spectrograph. Thus, the difficulties of focusing the radiation from the discharge to the spectrograph slot were excluded.

The lower flange of the reactor (I) incorporated three unions. One of them is used to effect evacuation of the system by the forevacuum pump, and the other two unions were connected via the straightway valves V_5 , V_6 and the metering valve VF with the gas-filled chamber CG and the mass-spectrometer MX-7301 (S_2) chamber, respectively. On the way to the forevacuum chamber, a metal four-joint was installed in the vacuum hose; one of its ends was connected with the vacuum gauge PD , and the other - with the T-joint put at the inlet of the metering valve. This set of connections ensured a constant control over the pressure in the reactor with both the inlet of the working gas and during its dissociation at discharge conditions. Besides, before each supply of a portion of the gas under study from the reactor to the mass-spectrometer, there was a possibility of passing the air once, twice or more through the measuring line followed by filling the line with the working product from the reactor chamber.

It should be noted that, in order to perform the mass-spectrometer analysis, the gas from the reactor

chamber was taken from its middle part. For this purpose, a copper pipe measuring $3 \cdot 10^{-3} \times 4 \cdot 10^{-3}$ m was placed in the reactor at a level of the discharge gap. The gas was directly supplied through the pipe to the admission device.

Since the products produced are mixed in the reactor exclusively due to heat convection, a miniature fan fed from the dc source B5-49 was mounted in order to enhance the efficiency of this process in the reactor. The fan was fixed so that its blades were at a level of the discharge gap. A possibility was also provided for gas blowing both along and across the discharge gap.

The forevacuum pump 2HBP-5DM (NI_2) and the diffusion turbomolecular pump VMN-500 (NR) were used to evacuate the mass-spectrometer.

The residual gas in the mass-spectrometer chamber was dried by heating the pickup (cylindrical head) of the analyzer for several hours using a specially manufactured electrical furnace. From the outside, the reactor chamber was cooled with a fan.

The discharge in the reactor chamber was initiated between the electrodes connected to the constant voltage source. The voltage at the supply source and across the electrodes (after R_{bal}) was measured by kilovoltmeters C-96 and C-196 (P_1 and P_2 , respectively, Fig. 2). The pressure in the mass-spectrometer chamber was measured with the use of the ionization vacuum gauge VIT-1 (PA). The thermal vacuum gauge (PT) was used to measure the residual gas pressure in the reactor chamber on pumping out. The working pressure of carbon dioxide in the reactor chamber was measured by means of the deformation vacuum gauge (PD) having the measuring range from 0.01 to 1.0 atm. The two-coordinate self-recorder N-306 (SR) registered the mass-spectra of initial and discharge-initiated products.

To perform experiments, it was necessary, first of all, that the mass-spectrometer be prepared for measurements. For this purpose, its operating volume was pumped down to the residual gas pressure $P \sim 1.5 \div 2 \cdot 10^{-7}$ torr; after that the mass-spectrum of the residual gas was registered. If this spectrum fully corresponded to the reference spectrum, the mass-spectrometer was considered to be in operable state. The inconsistency of the residual gas spectrum was mainly a result of an enhanced humidity, and was eliminated by heating the measuring pickup for several hours.

The plasma-chemical reactor was filled with the working gas in the following manner. The straightway valves V_1 , V_2 , V_3 , V_4 , V_6 were open with the valves V_5 and VF being closed. The working volume of the reactor was pumped to a pressure of 3 to 4 $\cdot 10^{-2}$ torr; after that the valve V_2 was shut off, and the valve V_5 was open. Then the carbon dioxide was admitted to both the reactor and the measuring duct to the pressure value required, the valve V_5 was shut off. With the use of the valve VF , CO_2 was supplied to the mass-spectrometer to a pressure of $\sim 2 \cdot 10^{-6}$ torr; the mass-spectrometer

was prepared for operation and the residual-gas mass-spectrum was registered by the self-recorder. This spectrum was compared to the one obtained previously. If there was a discrepancy between the spectra, the gas in the chamber was replaced by the method described above, i.e., pumping out the gas not corresponding to the standard and filling with a new portion. One of the examples of this discrepancy between the spectra can be the appearance of nitrogen N_2 in the spectrum, even a weak in-leakage of which (e.g., through a closed valve or the crack in the rubber tube of the measuring duct) is displayed on a mass-spectrogram. After taking the mass-spectrum of SO_x (the sensitivity of measuring devices was reduced in this case as compared to the case of taking the residual gas spectrum) the percentage content of obtained products was calculated with due account of the ionization cross sections.

At given conditions in the reactor (type of discharge, its parameters, pressure), a discharge was ignited for the assigned period of time (5, 10, 14, 30, 45 and 60 minutes). In this case, all the valves, except V3, were shut off.

Considering that a certain amount of SO_2 or a portion of previously produced gas remain in vacuum tubes, in order to obtain the true spectrum of products produced for the just another period of time, the measuring duct was pumped through twice or thrice each time before opening the metering valve, with a successive repetition of the following procedure.

With the valves V3, V5, V6 and V7 being shut off, the valves V1 and V2 were open and the system of the measuring duct was pumped through for 1 or two minutes. Then the valve V2 was cut off, and the valves V4 and V6 were open and the gas produced was let in, then the valve 6 was again cut off, the valve V2 was open, i.e., the system was pumped through.

After this process of pumping through the measuring system, the valves V2, V4 are shut off and the valve V6 is open. Then the metering valve VF is open, the gas produced is admitted into the mass-spectrometer and the spectrum is registered. Then, the taken spectrum was used to calculate the percentage content of gas components. The production of each component was determined by the difference between the percentage content of the corresponding gas components produced and the initial SO_2 . The thus obtained values corresponded to the content of components produced in the discharge.

It should be noted that for each subsequent time interval of product production in the discharge, the gas in the reactor chamber was replaced after prior evacuation of the products of the previous experiment.

To optimize the process of decomposition, a capacitor with a capacitance of ~ 150 to 1000 pF was connected in parallel to the discharge gap. The capacitance value determined the charge time and, correspondingly, the spark frequency in the discharge gap, $0.3 \text{ kHz} \leq f \leq 1.2 \text{ kHz}$. Besides, the spark

repetition rate was dependent on the interelectrode spacing and the power supply voltage.

3. METHODS AND DIAGNOSTICS

To determine the decrease in the SO_2 product under the discharge action, mass spectrograms taken before and after the discharge were processed. In this case, the peak amplitudes were corrected against the calibration gas Ar. A detailed analysis of the methods developed by us is given above.

First the corresponding peak amplitudes were determined (in mm) in the mass spectrograms. The difference D_i ($i = 1, 2, \dots$) describes the decrease in the i -th component of the gas mixture. The D_i/a_i value (a_i is the initial amplitude (in mm) of the i -th gas component) determines the relative decrease in the corresponding component. The total decrease in all gas components given by

$$\frac{D_S}{\sum a_i} = \frac{D_1 + D_2 + D_3}{a_1 + a_2 + a_3}.$$

Independently of the mass-spectrometry method of determining the decrease in gas components in the reactor, we have also used the method based on measurements of the total pressure and the average temperature of the gas mixture. Since the change in the reactor chamber pressure DP after the discharge action is determined as

$$DP = Dnk_B T + nk_B DT, \quad (8)$$

where n is the gas mixture density, T is the temperature of this mixture, k_B is the Boltzmann constant, the information on gas temperature variations is essential for elucidation of the contribution of each component in eq. (8) to the change in ΔP .

4. EXPERIMENTAL RESULTS

The processes of SO_x dissociation at atmosphere pressure in PCR were investigated during multi-spark discharge at voltage 12-13 kV, current 80 A, pulsed duration $0.8 \mu\text{s}$, repetition frequency 0.5-1.5 kHz.

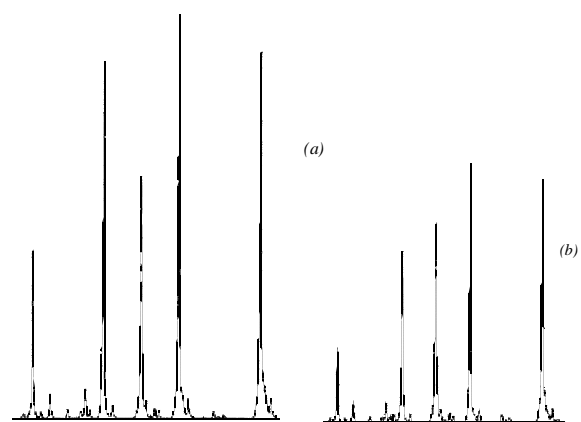


Fig. 4. Mass spectrograms before (a) and after (b) the discharge action.

To perform dissociation experiments we should previously produce the SO_x radicals. They were

obtained in a device described above by sulphur S burning in gases mixture of 80% O_2 and 20% Ar .

Fig. 4 shows mass spectrograms of the initial gas mixture and the gas mixture after a one-hour action of the discharge.

After determining the amplitudes of the corresponding components and equalizing these amplitudes against the calibration gas, we obtain the decrement (in percent) for these gas components:

$$\frac{DSO_2}{SO_2} = 19.75\%, \quad \frac{DSO}{SO} = 22.19\%, \quad \frac{DM_{32}}{M_{32}} = 41.77\%,$$

where M_{32} is the total amplitude of components with the mass number 32 (O_2^+ , SO_2^{++} , S^+), and DM_{32} is the decrement of this variable under the discharge action.

Then we have

$$\frac{D_S}{\sum a_i} = 22.7\%.$$

Similar measurements were also performed for 10, 20 and 30 minutes of the discharge action.

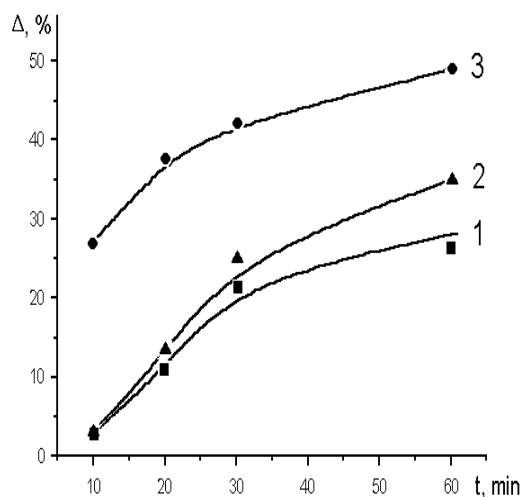


Fig. 5. Dependence of decrease DSO_2 - (1), DSO - (2), DM_{32} - (3) upon time

As is seen from Fig. 5, all the curves have the same behavior (each curve shows a trend to saturation).

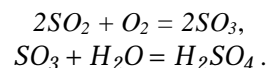
The experimentally measured temperature of gas mixture in the reactor on the 60th minute of discharge action exceeded the initial temperature by 10 K. It can be concluded that because the thermal contribution to the pressure variation makes $\frac{DT}{T} \sim 3.5\%$ (according to

(8), then $\frac{DP}{P} \approx \frac{Dn}{n}$, i.e., the pressure variation is mainly determined by the transition of components from the gas phase to the other.

The dependences of pressure and temperature growth of gas mixture upon time are shown in Fig. 6.

It should be also noted that as early as in a few minutes after the discharge ignition the surface of the lower electrode showed a wet spot that could be attributed to the production of SO_3 in the discharge.

Typically, in the presence of even an insignificant percent of water SO_3 tends to form sulfuric acid:



After the gas produced in the discharge was investigated, the reactor was degassed. On opening the reactor chamber, an acid residue could be seen on the reactor walls. This residue actively reacted with a litmus paper.

5. SUMMARY

Experiments have demonstrated that the plasma-

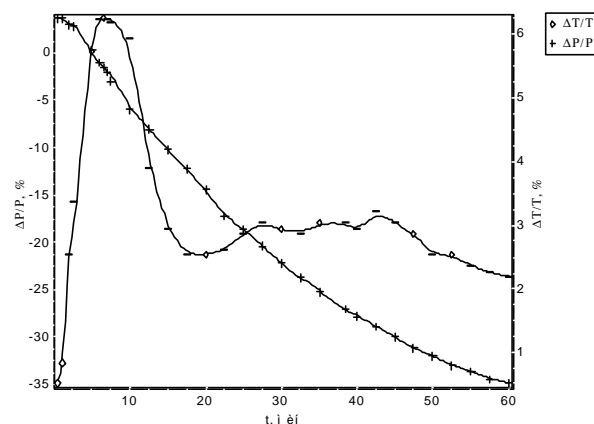


Fig. 6. Relative change of gas mixture pressure and temperature versus time.

chemical reactions in the travelling spark discharge are responsible for the effective dissociation of SO_2 with a crystalline precipitate. The value 22.7% of SO_x decrease was evaluated from the mass-spectra of initial mixture and treated one by the discharge during one hour

Two techniques were developed and used to determine the decrease in the SO_2 product and the accompanying gas substances: (1) from change in SO_2 peak amplitudes in mass spectrograms before and after the discharge, and (2) from variations in the pressure and temperature of the whole gas mixture in the reactor during the discharge. It is shown that the decrease in pressure is determined by the fact that gas mixture components cease to be in the gas phase.

The undertaken investigations give evidence to assume that the process of SO_2 dissociation in our discharge can be optimized by accumulating the decay products.

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