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THEORETICAL ANALYSIS AND EXPERIMENTAL STUDY OF H_2S DISSOCIATION PROCESSES IN ULTRAHIGH-FREQUENCY PLASMOTRON

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Theoretical analysis of aerodynamic conditions in a plasma chemical reactor with tangential gas supply is carried out. It is shown that due to the creation of a swirling flow in the reactor there is a pressure gradient, due to this along the vertical axis there is a vacuum zone, which contributes to the occurrence of plasma discharge. On the basis of the carried-out experimental researches of plasmolysis of hydrogen sulphide in a swirling stream and the analysis of images of the plasma discharge with use of monochromatic light filters the general structure of the plasma discharge is established. The influence of the temperature gradient in the reactor on the possibility of the formation of sulphur clusters as a prerequisite for the formation of a high molecular weight product – polymeric sulphur – was established.

Key words: superhigh-frequency plasma; plasma discharge; plasma chemical reactor; waveguide; hydrogen sulphide; decomposition; dissociation; hydrogen; sulphur.

Introduction

Hydrogen sulphide gases are formed in the processes of purification of natural gas from H_2S, desulphurization of oil, in the processes of coking of coal. Previous studies have shown that one of the most promising methods is plasma chemical processing of hydrogen sulphide gases [1]. This allows to obtain two valuable products: hydrogen (it is the basis of hydrogen energy [2], used in the chemical and petrochemical industries [3]) and sulphr in the form of rhombic (used in the production of sulphuric acid, in the rubber industry, agriculture) or polymer [4] (mostly used as a vulcanizer) modifications.

Plasma-chemical processes are highly energy-intensive, as the amount of energy introduced per unit volume of the reactor is several orders of magnitude higher than in traditional chemical reactors. To be implemented in industrial conditions, they require in-depth study both in terms of process chemistry and mass and energy transfer processes, aerodynamics, and so on. It is very important to ensure the stability and reliability of the chemical plasma reactor. Although the process of plasma chemical treatment has been tested in industrial conditions [1], a number of issues are still insufficiently studied. This applies, in particular, to the aerodynamic conditions in an ultrahigh-frequency (UHF) plasma chemical reactor.

Analysis of information sources

Analysis of real plasma chemical processes in a plasma chemical microwave reactor is a rather difficult technical task, as there are problems with measuring the concentration profile of environmental components, pressure and temperature gradients in the radial and axial directions. Therefore, to analyze of plazma chemical processes numerical modeling methods are mostly used [5].

Bogdanov and others performed a set of studies of plasma-chemical processes involving oxygen [6-8], on the basis of which they created appropriate mathematical models. It has been shown that the processes in oxygen plasma significantly depend on the parameters of the medium and the energy supplied to the plasma chemical system. The results of studies of processes involving weakly ionized plasma in vortex flows are given in [9], and aerodynamic processes in barrier discharge plasma are given in [10]. It is shown that in the zone of aerodynamic vacuum there is a probability of occurrence and stabilization of plasma discharge. The dependence of the plasma structure on the aerodynamic parameters of the flow is given in [11]. During the studies of the structure of the plasma discharge, the presence of a surface layer was detected. It was studied in various gaseous media, in particular in air [12] in barrier discharge plasma. It was found that the interaction of the gas flow with the weakly ionized near-surface plasma occurs with the participation of the surface layer [13].

Stationary and nonstationary methods were used to measure the flow rate where barrier discharge plasma was initiated [14]. The dependence of the characteristics of the glow discharge at atmospheric pressure on the energy and aerodynamic parameters of research has been experimentally established [15].

The study of the dynamics of ultrahigh-frequency nonequilibrium plasma excited in a nitrogen medium was carried out on the basis of the analysis of ultraviolet radiation generated in this case [16].

However, most of the studies concerned barrier or corona-discharge plasma

The aim of the study: analysis of the system of aerodynamic and chemical processes in direct-flow and swirling streams of ultrahigh-frequency plasma, in particular, in the decomposition of hydrogen sulphide.

Materials and methods of research

Experimental studies of plasma-chemical processes in ultrahigh-frequency plasma in hydrogen sulphide were performed on a laboratory installation consisting of the following main elements: microwave radiation generation unit (maximum power 2.5 kW, microwave radiation frequency 2.45 GHz, plasma chemical reactor with waveguide) and the hydrogen sulphide generation unit [17]. Hydrogen sulphide was generated by reacting 20 % H_2SO_4 solution with Na_2S solution. Analysis of the gas phase for hydrogen sulphide content was performed chromatographically ("Gasochrome 3101"). Before being fed to the plasma chemical reactor, the hydrogen sulphide gas was dried with calcium chloride.

The dependence of the shape and geometric dimensions of the plasma discharge on pressure, gas flow rate, microwave radiation power was studied on the basis of analysis of numerous photographic images made using monochromatic light filters (blue, red and orange). Each of these filters transmits light with a certain wavelength, ie with certain energy. This corresponds to the temperature of the plasma components. The use of different light filters allows to establish the general structure of the plasma discharge. The temperature of the gas stream at the outlet of the reactor was measured using a thermocouple.

The content of polymeric modification in sulfur, which was formed due to the decomposition of hydrogen sulfide, was determined by weight method after extraction of soluble (rhombic) modification of sulphur with toluene.

Research results and their discussion

Theoretical analysis of processes in a plasma chemical reactor

The decomposition of hydrogen sulphide in ultrahigh-frequency plasma can be described by the general equation

$$H_2S \Leftrightarrow H_2 + S. \quad (1)$$

The hydrogen sulphide molecule is "transparent" for ultrahigh-frequency radiation. Therefore, under normal conditions, it practically does not interact with microwave electromagnetic radiation. Therefore, to ensure the absorption of this radiation, it is necessary to create conditions under which the medium becomes electrically conductive. This is achieved by ionization of hydrogen sulphide molecules, due to the ionization of plasma.

The main condition for the occurrence of plasma is inequality

$$E_{UHF}/V \times N \geq E_i, \quad (2)$$

here E_{UHF} – the value of the energy introduced into the elementary volume of the plasma chemical reactor, kJ/mol; V – the elementary volume of the reactor, dm^3; N – the number of H_2S molecules in the elementary volume, mol; E_i – ionization energy of H_2S, kJ/mol.

This condition can be achieved in the following ways:

1) increasing the power of microwave radiation with axial supply of gas flow;

2) reducing the pressure in the plasma chemical reactor (up to 5–10 Pa);

3) providing a significant pressure gradient in the radial direction of the reactor due to the tangential supply of gas to the reactor;

4) combining the above methods.

Increasing the power of microwave radiation requires a simultaneous significant increase in gas consumption. As the gas flow rate increases, the flow rate will increase, which is known to reduce the pressure. In this case, the concentration of hydrogen sulphide will decrease and at a certain gas consumption there will be conditions that will satisfy the conditions of inequality 1.

Reducing the pressure in the plasma chemical reactor makes it possible to achieve the condition of inequality 1. However, the gradient of hydrogen sulphide concentrations in the radial direction is practically absent. Therefore, the area of existence of the plasma discharge will be limited by the physical size of the reactor, in particular the diameter. This can lead to the localization of plasma on the reactor wall from the side of the introduction of microwave energy and the destruction of the reactor.

Tangential gas supply to the reactor leads to the appearance of radial and tangential components of the flow rate (Fig. 1).

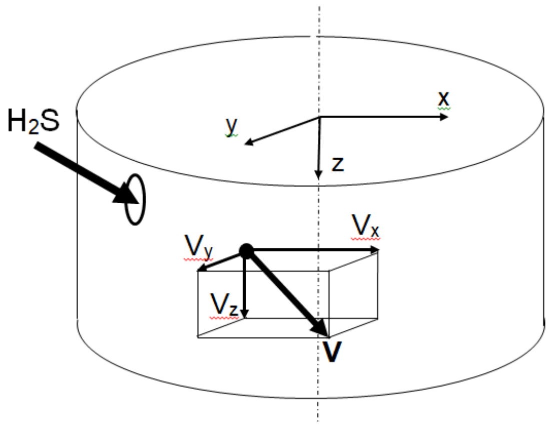

Fig. 1. Scheme of rates distribution in a swirling flow in plasma chemical reactor:
V_x – radial; V_y – tangential; V_z – axial; V – resulting

The ratio of the tangential component of the velocity to the axial (V_y/V_z) determines the degree of twisting of the gas flow. If the axial component of the velocity (V_z) is much larger than the other two, which in this case are neglected, the flow is slightly twisted, ie it is almost direct. In this case, the operation of the reactor is close to the model of ideal displacement (RID). The velocities of all flow particles will be almost the same (the wall effect is neglected), and their concentration profile in the cross section of the reactor will be constant.

Due to the action of centrifugal forces in the swirling flow relative to the vertical axis of the cylindrical reactor there is a vacuum, and on the periphery (near the reactor wall) – pressure increase, ie there are pressure gradients and concentrations of plasma-forming gas particles.

Quantitative assessment of the intensity of the twisting flow in the reactor is determined by the twisting parameter Q_{dtf}.

$$Q_{dif} = \frac{M}{K \cdot r_0}, \tag{3}$$

here M – the moment of the amount of movement of the jet; K – the amount of movement of the jet; r_0 – the radius of the jet, which under extreme conditions is equal to the radius of the cylindrical part of the plasmatron.

For a cylindrical jet, the values of M and K are determined by the equations

$$M = 2\pi\rho \int_0^{r_0} r^2 V_x V_y dr, \tag{4}$$

$$K = 2\pi \int_0^{r_0} r(\rho V_x^2 + P) dr, \tag{5}$$

here r – the density of the gas phase at a certain point in the reaction space of the plasmatron; r – current value of radius.

In the partial case, the degree of torsion of the flow can be estimated from the maximum values of the tangential and axial velocity components, which are easiest to determine experimentally under normal conditions without plasma excitation. According to the results of the analysis of the temperature field in the reactor, the components of the gas flow rate are easy to calculate because they are proportional to the volumetric flow rate of the gas, which increases in proportion to the temperature. The degree of twisting of the flow in the reactor in the cross section of the waveguide is equal to

$$\eta_3 = \frac{V_{y,\max}}{V_{x,\max}}. \tag{6}$$

Expression (6) is directly related to equation (3) in the case where the gas rotates as a conditionally solid. A priori, it can be predicted that in a swirling stream under the action of centrifugal forces, the decomposition products of hydrogen sulphide will be separated: heavier (sulphur) will be carried to the periphery, and lighter (hydrogen) will move to the center due to a certain vacuum. Therefore, equation (6) can describe the twisting of sulphur particles (clusters), which under certain conditions will form a condensed phase.

In this case

$$V_y = \frac{V_{y,\max} r}{r_0}, \tag{7}$$

$$V_x = V_{x,vax}. \tag{8}$$

From here,

$$M = 0,5\pi\rho V_{x,\max} V_{y,\max} r_0^3, \tag{9}$$

$$K = \frac{\eta_K}{2(1-0,25\eta_K^2)}. \tag{10}$$

Twisting of the flow, which will obviously lead to a change in pressure in the reactor in the radial direction, which in general can be expressed

ву

$$\frac{\partial P}{\partial r} = \rho \frac{\partial^2 V_y}{r}. \tag{11}$$

Quantitatively, the change in pressure over the radius of the reactor can be expressed by our derivation on the basis of the known dependence, which is a hydrodynamic model of a microwave plasmatron

$$dP = \frac{P_0}{(1-(K-1)\rho_0 I_0 (8KP_0\pi^2 r^2)^{-1})^2} dr, \tag{12}$$

here P, P_0 – pressure according to the radius of the reactor r (m) and the initial, Pa; K – the amount of movement of the jet; ρ_0 – the initial density of the medium, kg/m^3; I_0 – the intensity of the swirling flow.

$$K = 2\pi \int_0^r r(\rho \cdot u_z^2 + P) dr, \tag{13}$$

$$I_0 = \frac{u_y}{2\pi r}. \tag{14}$$

Analysis of equation (12) shows that the pressure decreases with decreasing radius, reaching a minimum value in the axial part of the reactor. It is obvious that in the axial zone of the reactor $(r \to 0)$ there are the most favorable conditions (conditions are fulfilled, according to inequality (2)) for the occurrence of a plasma discharge, and in the peripheral regions – the contrary, it is impossible.

The existence of a plasma discharge in the central (axial) region of the reactor obviously leads to the presence of a region of high temperature. In the reactor zone, where the existence of a plasma discharge is unlikely or impossible, the temperature is much lower. Thus, the tangential introduction of the gas flow also causes a radial temperature gradient.

Simultaneously with the increase in radius, ie at the periphery of the reactor, the pressure increases. It is obvious that sulphur particles are transferred to the periphery, so the pressure of sulphur vapors will increase in the peripheral regions of the reactor, and, consequently, there may be conditions under which condensation of sulphur vapors will occur with the formation of polyatomic sulphur molecules.

To establish the possibility of supersaturation of sulphur vapor and, accordingly, the possibility of condensation of sulphur vapor, we assume that the system may have reversible reactions with the formation of the starting material – hydrogen sulphide by reaction (1). Therefore, it is necessary to compare the partial pressure of sulphur vapor over its melt (P_P) and the equilibrium vapor pressure of sulphur P_S. The equilibrium constant of reaction (1), expressed through the activity of the components of the gas phase

$$K = \frac{a_{H_2S}^2}{a_{H_2}^2 \cdot a_S} \tag{15}$$

is described by the temperature dependence

$$lgK = 8369/T - 3.84T + 0,606 \times 10^{-3} T + +0.066 \times 10^5/T^2 + 6.824. \tag{16}$$

The activity of the gas phase component is proportional to its partial pressure $a_i = P_i / P_o$, here P_o =1,013 ×10^5 Па. i Therefore, equation (15) will look like

$$K = \frac{P_{H_2S}^2 \cdot P}{P_{H_2}^2 \cdot P_S}. \tag{17}$$

According to the stoichiometry of reaction (1), $P_{H_2} = 2P_S$. Given this, we get

$$K = \frac{P_{H_2S}^2 \cdot P_0}{4P_S^3}. \tag{18}$$

From here, the known values of K for each given value of the partial pressure of hydrogen sulphide can be used to calculate the vapor pressure of sulphur P_S, which is established by the equilibrium of the chemical reaction (1). For example, for partial pressure $P_{H_2S} = 0.1P_0 = 1.013 \cdot 10^4$ Pa, we get the expression

$$P_S = 0.1P_0(2{,}5/K)^{1/3}. \qquad (19)$$

The results of calculations performed by equation (19) taking into account equation (16) show that with increasing pressure in the reactor and increasing temperature, the partial pressure of sulphur, which is set at equilibrium, approaches the value of liquid sulphur vapor elasticity, and therefore condensation becomes more likely sulphur vapors and the formation of sulphur clusters.

The vaporr pressure over liquid sulphur (P_{liq}) can be determined by the equation of West and Menzies

$$lnP_{liq} = 25{,}45675 - 9414{,}203/T. \qquad (20)$$

Sulphur pairs are heterogeneous in molecular composition and are mostly in the form of four molecular forms: S_8; S_6; S_4; S_2.

The equilibrium composition of sulphur vapors can be determined taking into account the equilibrium constants of three independent reactions:

$$\begin{aligned} &1)\ S_4 = 2S_2; && K_1 = (P_2)^2/P_4; \\ &2)\ S_6 = 3S_2; && K_1 = (P_2)^3/P_6; \qquad (21) \\ &3)\ S_8 = 4S_2; && K_1 = (P_2)^4/P_8. \end{aligned}$$

According to the experimental data of Brown, Peter and Neveling, the temperature dependence of the equilibrium constants K_1, K_2 and K_3 is described by equations

$$lhK_1 = -272739.68/PT + 27.5771; \qquad (22)$$
$$lhK_2 = -611839.62/PT + 58.0791; \qquad (23)$$
$$lhK_3 = -885251.5/PT + 85.5181. \qquad (24)$$

By the values of the equilibrium constants according to the system of equations (22)–(24), we obtain three equations with four unknowns. Since we are interested in pairs in equilibrium with liquid sulphur, we have another connection equation in the form

$$P_2 + P_4 + P_6 + P_8 = P_{liq}. \qquad (25)$$

Solving together (22)–(24) and (25), we obtain a polynomial with one unknown

$$(P_2)^4 + K_2^{-1}K_3(P_2)^3 + K_1^{-1}K_3(P_2)^2 + $$
$$+K_3(P_2) + K_3P_{liq} = 0. \qquad (26)$$

For each temperature by equations (16) and (17) calculated the values of P_{liq}, K_1, K_2, K_3, and then found the root of the polynomial (26), which satisfies the condition $P_{liq} > P_2 > 0$. Next, using (20), calculated the partial pressures P_4, P_6 and P_8. The results of the calculations are presented in Fig. 2.

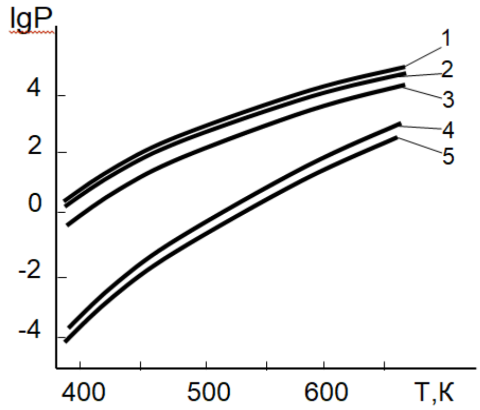

Рис. 2. The dependence of vapor pressure over the melt of sulphur (1) and the partial pressures of different molecular forms of sulphur: S_8 – 2; S_6 – 3; S_4 – 4; S_2 – 5

These calculations show that as the temperature of the plasma chemical reactor decreases, there are real conditions for the formation of a condensed phase in the form of sulphur microdroplets.

However, it should be borne in mind that in a plasma chemical reactor due to the dissociation of hydrogen sulphide

$$H_2S\ \hat{U}\ 2H^{\cdot} + S^{\cdot} \qquad (27)$$

products of a radical nature are formed, namely the hydrogen radical and the sulphur radical.

Under the action of centrifugal force, sulphur as the heaviest of the products diffuses to the periphery of the reactor. As the distance from the reactor axis increases, the concentration of sulphur particles (atoms, radicals, etc.) increases. Together with a decrease in temperature in the radial direction, this should promote the formation of polyhydric sulphur compounds. primarily due to the processes of recombination of sulphur radicals with the formation of neutral molecules and products-biradicals, for example:

$$S^{\cdot} + S^{\cdot} = S_2;$$
$$nS^{\cdot}\ ®\ {}^{\cdot}S\text{–}S_{n-2}\text{–}S^{\cdot}.$$

In turn, this will increase the effect of centrifugal force. Thus, due to the system of these processes, the number of sulphur atoms in the product increases, which can play the role of the nucleus of a high molecular weight product – polymeric sulphur.

Experimental study of the influence of plasma discharge parameters on plasmolysis of hydrogen sulphide

It was theoretically shown above that the tangential supply of gas to the reactor should ensure the occurrence of a plasma discharge in the central region of the reactor. To confirm this, experiments were performed to investigate the effect of conditions on the occurrence of plasma discharge and its stability.

Axial gas supply, as predicted theoretically, makes it possible to create a plasma discharge at a microwave power of 1.1 kW and a flow velocity of more than 0.32 m/s. However, the plasma discharge is diffuse (Fig. 3, *a*) and occupies almost the entire volume of the rector in the area of its location in the microwave waveguide. This causes its instability in space. Due to this, the plasma is localized on the reactor wall by the introduction of microwave energy (Fig. 3, *b*). As can be seen, the glow intensity of the entire reactor in the waveguide region increases sharply. It increases to 1500 °C and more. As a result, the destruction of the reactor is possible.

Plasma discharge occurs in the central zone at a tangential gas supply with a velocity of at least 0.25 m/s, even at atmospheric pressure at the reactor inlet and radiation power of 0.65 kW. It is contracted, ie characterized by the presence of fairly clear contours, in particular in the waveguide (Fig. 4).

Analysis of numerous images of the plasma discharge, which occurred after the tangential introduction of hydrogen sulphide, made it possible to clearly establish the structure of the plasma and its relationship with the position of the discharge relative to the waveguide (Fig. 4).

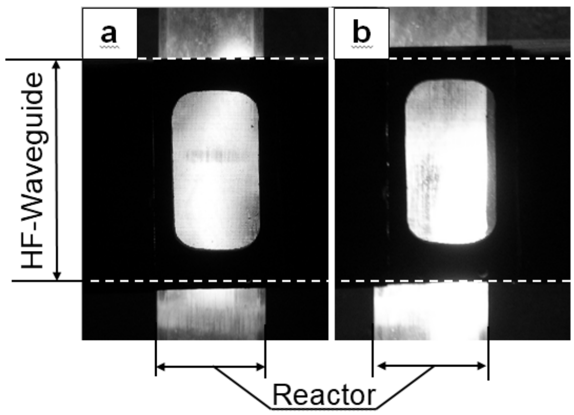

Fig. 3. Photo of microwave discharge with axial gas supply: a – diffuse region of plasma after its initiation; b – localization of the plasma discharge on the reactor wall after some time

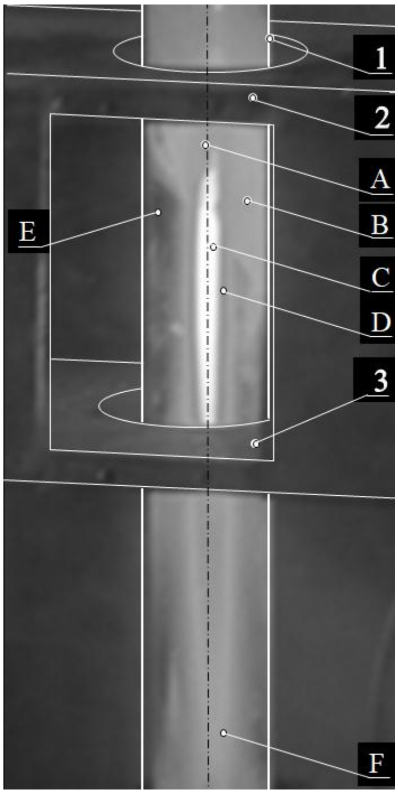

Fig. 4. Photo of the plasma microwave discharge in the microwave reactor (the contours of the reactor elements are shown in white lines): 1 – reactor; 2 – microwave path (waveguide); 3 – "window" in the waveguide; A – the axis of the reactor; B – "hot" swirling flow; C – contracted plasma discharge; D – transition zone; E – "cold" area; F – the diffuse zone of the discharge

On the image of plasma discharge clearly shows the flow twisting zone (area *B*). It is located in the upper part of the waveguide. The temperature in this area reaches 600–700 °C. such a rather high temperature is due to the presence of circulating vertical flows in plasma chemical reactors.

It is the twisting of the flow that causes a vacuum zone along the reactor axis and the appearance of a contracted plasma discharge in it (region *C*). At a gas speed of 0.25 m/s and a power of 1.1 kW, the length of the discharge is 5–6 cm, and the diameter is not more than 1 cm. Therefore, this area is called a "spoke". Throughout the study, the situation in this area remains stable. This zone is characterized by the presence of a transition region *D*. Through this region there is a mass transfer between the zone of plasma existence and the plasmaless medium. It was found that slight fluctuations are possible in this zone.

The displacement of the region of existence of the discharge downward relative to the upper part of the waveguide is due to the presence of the axial component of the flow velocity.

The position of the plasma discharge significantly depends on the parameters of the process (but the shape of the discharge does not change – it remains clear, contracted). For example, with a decrease in velocity to 0.2 m/s, as well as with an increase in the power of microwave radiation to 1.4 kW and more, the discharge is shifted upward. With an increase in gas velocity over 0.3 m/s and with an increase in microwave power over 1.4 kW, the discharge "stretches" to 25–30 cm: it begins above the waveguide and ends far below it.

The region E is characterized by the lowest temperature in the reactor in the area of its location in the waveguide (about 350–400 °C). The presence of such zones with a lower temperature can be explained by the imperfect twisting of the gas flow with a small number of nozzles for tangential gas supply (in this case is 2). In the case of increasing the number of nozzles through which gas is fed into the reactor, up to 4 area F exists only in the upper part of the reactor.

At a distance of 4–10 cm from the down part of the waveguide (it depends on the gas velocity and the power of microwave radiation), you can select the area F. In it, the area of hot gas flow expands and oscillates (500–700 °C). This is due to the redistribution of the velocity components in the lower part of the reactor: the radial decreases and the axial component increases accordingly.

It is established that aerodynamic conditions significantly affect the degree of decomposition of hydrogen sulphide. Thus, at a microwave radiation power of 1.1 kW and a hydrogen sulphide flow rate of 0.32 m/s, the degree of plasmolysis of hydrogen sulphide was equal to: at axial feed – 58 %; for tangential submission – 87 %. In the first case, the gas flows directly through the plasma. Because of this, the time spent in the plasma discharge zone is about 0.25 s. In the second case, due to the presence of radial and axial components and the existence of a pressure gradient in the reactor, there is a circulation of unreacted hydrogen sulphide in the radial direction. Due to this, hydrogen sulphide periodically enters the zone of existence of the plasma discharge during the movement in the reactor. Due to this, the degree of decomposition of hydrogen sulphide is much higher than with axial gas supply. The content of polymeric sulphur in the product obtained at the rate of cooling of the gas at the outlet of the reactor about 80 K/s increased from 40–50 (for for tangent feed) to 90–97 %.

Conclusions

The possibility of purposeful localization of plasma discharge along the vertical axis of the plasma plasma reactor due to tangential gas introduction is theoretically shown and experimentally confirmed. This ensured the stability of the plasma discharge and the stability of the plasma chemical decomposition of hydrogen sulphide in a fairly wide ranges of linear gas velocities and microwave power.

Tangential supply of gas to the reactor contributes to a significant increase in the degree of plasmolysis of hydrogen sulphide due to its circulation in the radial direction.

Due to the action of centrifugal force there are prerequisites for the formation of polyhydric sulphur clusters as nuclei of the condensed phase in the form of polymer modification of sulphur.

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ТЕОРЕТИЧНИЙ АНАЛІЗ ТА ЕКСПЕРИМЕНТАЛЬНЕ ДОСЛІДЖЕННЯ ПРОЦЕСІВ ДИСОЦІАЦІЇ H_2S У НАДВИСОКОЧАСТОТНОМУ ПЛАЗМОТРОНІ

Виконано теоретичний аналіз гідродинамічних умов у плазмохімічному реакторі за тангенціального подавання газу. Показано, що внаслідок створення закрученого потоку в реакторі виникає градієнт тиску, завдяки цьому вздовж вертикальної осі формується зона розрідження, що сприяє виникненню плазмового розряду. На підставі експериментальних досліджень плазмолізу сірководню у закрученому потоці та аналізу зображень плазмового розряду із використанням монохроматичних світлофільтрів визначено загальну структуру плазмового розряду. Встановлено вплив градієнта температури в реакторі на можливість формування кластерів сірки як передумови утворення високомолекулярного продукту – полімерної сірки.

Ключові слова: надвисокочастотна плазма; плазмовий розряд; плазмохімічний реактор; хвилевід; сірководень; розклад; дисоціація; водень; сірка.