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## WATER VAPOR CONVERSION DURING THE INTERACTION BETWEEN AN EVAPORATED HYDROGEN FLUORIDE SOLUTION AND CARBON IN THE FILTRATION COMBUSTION MODE

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The results of laboratory studies of the water vapor conversion when an evaporized aqueous solution (EAS) of HF and oxygen is being fed into a stationary layer of granular graphite have been presented. It was established that the characteristic time for the water vapor conversion upon the contact of the EAS of HF with carbon at a temperature of about 1500 K was 10 s. Comparison of the experimental results with the literature data on high-temperature interaction of water vapor and carbon showed that HF had little or no effect on the rate of this interaction at a temperature of about 1500 K. Our method derived from the high-temperature interaction of the EAS of HF with carbon can serve as the basis of an industrial technology for the dehydration of an aqueous solution of HF, including azeotropic one.

**Keywords:** hydrogen fluoride, hydrofluoric acid, water gas, filtration combustion

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## КОНВЕРСИЯ ВОДЯНОГО ПАРА ПРИ ВЗАИМОДЕЙСТВИИ ИСПАРЕННОГО РАСТВОРА ФТОРИДА ВОДОРОДА С УГЛЕРОДОМ В РЕЖИМЕ ФИЛЬТРАЦИОННОГО ГОРЕНИЯ

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Приведены результаты лабораторного исследования конверсии водяного пара при подаче испаренного водного раствора (ИВР) фторида водорода HF и кислорода в стационарный слой гранулированного графита. Установлено, что характерное время конверсии водяного пара при контакте ИВР HF и углерода при температуре около 1500 К составляет величину порядка 10 с. Сравнение результатов эксперимента с литературными данными по высокотемпературному взаимодействию водяного пара и углерода показало, что при указанной температуре присутствие HF практически не влияет на скорость взаимодействия водяного пара с углеродом. Метод, базирующийся

на высокотемпературном взаимодействии ИВР HF с углеродом, может служить основой промышленной технологии дегидратации водного раствора HF, в том числе азеотропного.

**Ключевые слова:** фторид водорода, плавиковая кислота, водяной газ, фильтрационное горение

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### Introduction

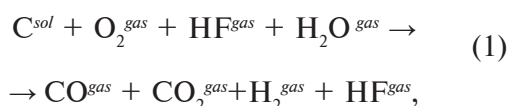
Aqueous solution of hydrogen fluoride HF (hydrofluoric acid) is produced by a number of industries; for example, in processing of depleted uranium hexafluoride  $UF_6$  by hydrolysis [1, 2].

Anhydrous hydrogen fluoride (aHF) [3, 4] is mainly used in industry as a fluorinating agent; its water content should be no higher than 0.06% [5]. For this reason, developing technologies for producing anhydrous hydrogen fluoride from its aqueous solutions is an important task.

While aHF is typically obtained from its aqueous solution by distillation [5], this solution is azeotropic with a hydrogen fluoride content of about 35–40% [6], so the mixture cannot be separated into individual components by conventional distillation. An azeotrope is one of the components with any composition of the initial mixture subjected to distillation. Thus, it is impossible to completely remove hydrogen fluoride from its aqueous solution by distillation.

Different studies considered dehydration of hydrofluoric acid with sodium fluoride [7] and concentrated sulfuric acid [8]. The first method has no industrial application at present. As for the second method, 75% sulfuric acid in the amount of about 30 kg per 1 kg of aHF is formed as a by-product, so it is difficult to select structural materials for industrial implementations of this method.

Ref. [9] proposed a method for dehydration of an aqueous solution of hydrogen fluoride, including azeotropic, by high-temperature interaction of water vapor with carbon with filtration combustion by the scheme:



where the superscripts *sol* and *gas* correspond to the solid and gas phase of the substances.

Using thermodynamic calculations and laboratory studies, Ref. [9] confirmed that hydrogen fluoride is the only fluorine-containing

substance in the system at temperatures above 1000 K.

Thus, it was established that process (1) is in principle possible if hydrogen fluoride remains inert with respect to other components of the system, and water vapor interacts with carbon to form hydrogen  $H_2^{gas}$  and carbon monoxide  $CO^{gas}$ .

Interaction of water vapor with carbon is well studied in theory on gasification of solid fuels. In particular, it was found in [10] that the characteristic time of interaction of water vapor with carbon of different grades is several seconds at 1500–1600 K.

However, we have not uncovered any data in literature on the reaction rate for interaction of water vapor with carbon in the presence of hydrogen fluoride. It is known that hydrogen fluoride is a polar substance (its dipole moment is equal to 1.88 D) and, as such, has catalytic and inhibitory effects on many chemical processes [4].

The goal of this study has consisted in determining the depth of conversion of water vapor during high-temperature interaction of vaporized aqueous solution of hydrogen fluoride with a layer of granular graphite when oxygen is fed into the layer.

### Characteristics of the initial materials

We used ground artificial graphite GII-A (TU 1916-109-71-2000) with a bulk weight of 840 kg/m<sup>3</sup> and the content of the basic substance no less than 99% for the laboratory experiments; the graphite had ash content of no more than 1%, moisture and sulfur contents not more than 1.0 and 0.05%, respectively. The granulometric composition of the mixture is given in Table 1.

The composition of impurities was detected using ICPE-9000, an inductively coupled plasma emission spectrometer, and FTIR-8400S, a FTIR spectrometer. The results of the study suggest that the main impurities in the initial graphite were calcium and iron silicates.

Table 1

## Granulometric composition of original graphite

Sieve mesh size, mm	10	7	5	2.0	1.4	0.40	0.25
Weight content of graphite in mixture, %	0.0	0.8	20.0	63.7	12.6	2.9	0.0

It was found by leaching the graphite with 0.4% sodium hydroxide that the acid component was absent in the initial graphite.

We used aqueous solutions of hydrogen fluoride with the weight contents of hydrogen equal to 40% and 72%, since these solutions are formed in a number of technological processes [1, 2].

#### Laboratory setup and experimental procedure

We constructed a laboratory setup for experimental studies on high-temperature interaction of water vapor and carbon in the presence of oxygen and hydrogen fluoride. The schematic of the setup is shown in Fig. 1.

The steel cylindrical reactor *I* with a volume of 23 L, a height of 400 mm and an internal diameter of 273 mm is equipped with a cooling jacket and systems for supplying components and removing reaction products.

The temperature of the coolant in the cooling jacket was 370 K.

A thick-walled copper nozzle *2* with a diameter of 28 mm and a wall thickness of 8 mm was used to supply oxygen into the layer of granulated carbon and evaporated aqueous solution of hydrogen fluoride. A cooling jacket was installed to cool the nozzle in its upper part; water was supplied into the jacket at a temperature of 370 K. Experiments were carried out with two positions of nozzle *2* in reactor *I*.

In position I (Fig. 2, *a*), the nozzle was installed in the side outlet of the reactor's upper flange at an angle of 45°, with the nozzle shut-off valve located on the vertical axis of the reactor. In position II (Fig. 2, *b*), the nozzle was installed in the central outlet of the upper flange, and the reactor was turned over.

The temperature distribution in the zone where gas flowed out of the nozzle was monitored using chromel-alumel thermocouples  $T_1-T_4$

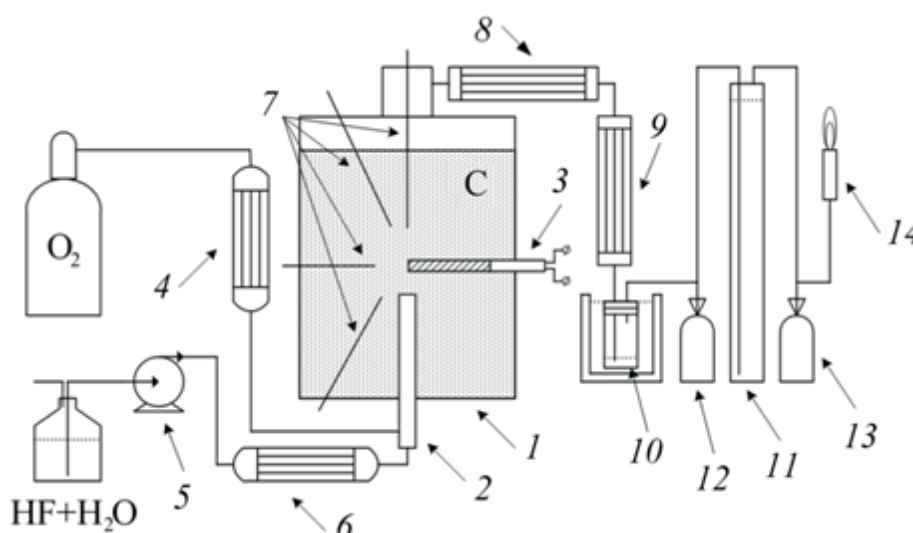


Fig. 1. Layout of laboratory setup:

- reactor *I*; two-component nozzle *2*; removable electric heater *3*;
- heat exchanger *4* for heating  $O_2$ ; peristaltic pump *5*;
- heat exchanger *6* for evaporating and overheating aqueous solution of HF;
- thermocouples *7*; nickel heat exchangers *8*, *9*;
- acid collector *10*; alkaline absorber *11*; nickel tubes *12*, *13*;
- device *14* for combustion of  $H_2$  and CO in air oxygen

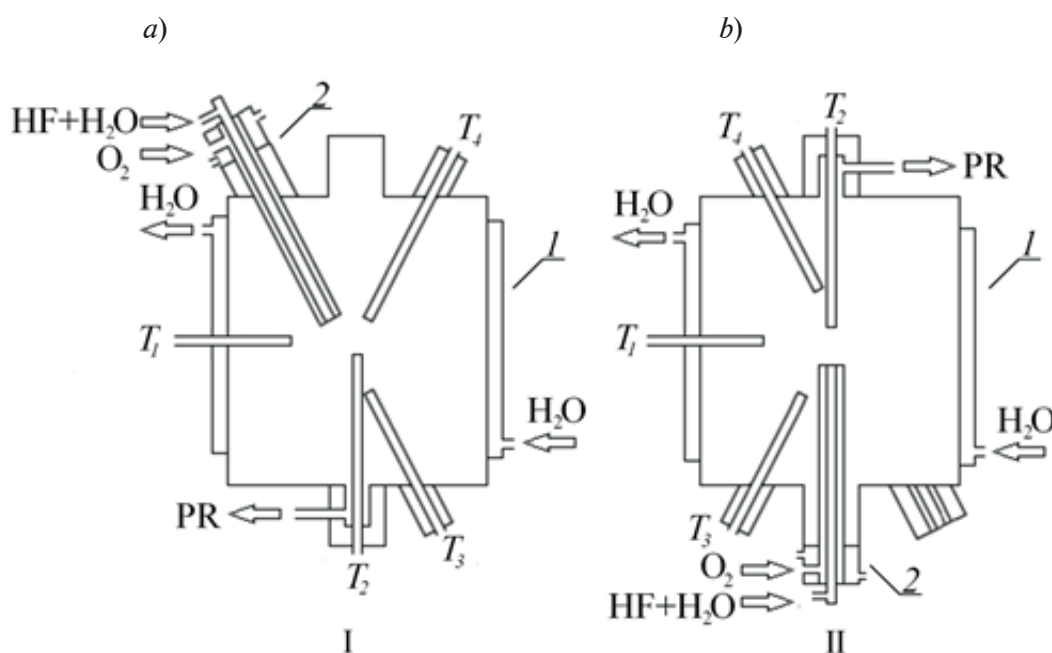


Fig. 2. Schematics for two layouts of nozzle (2) and thermocouples ( $T_1-T_4$ ) in reactor (I); a, b are positions I and II; PR are products of reaction

installed in nickel covers with a diameter of 6 mm and a wall thickness of 1.5 mm.

At the beginning of the experiment, granular graphite was loaded into the reactor up to the level of the upper lid, while the average mass of carbon backfill was 16 kg. Next, the carbon layer was preheated using a removable electric heater 3 (see Fig. 1) installed near the nozzle shut-off valve. After the graphite near the nozzle was heated to a temperature of 900 K, the electric heater was removed from the reactor and oxygen was supplied through the nozzle to further heat the carbon layer and maintain the temperature above 1000 K. In order to prevent condensation of water vapor and hydrogen fluoride on the inner surfaces of the nozzle, oxygen was heated to 500 K in heat exchanger 4 before it was fed into the reactor.

Next, a pre-evaporated aqueous solution of hydrogen fluoride was supplied into the heated carbon layer together with oxygen. The solution was dispensed with peristaltic pump 5 and evaporated in heat exchanger 6 at a temperature of 600 K.

The boundaries and characteristic dimensions of the region in the graphite layer where the temperature exceeded 1000 K were estimated using the thermocouples. Notably, the equilibrium in reaction (1) is

shifted to the right at this temperature.

To cool the gas flow of reaction products, nickel heat exchangers 8 and 9 (see Fig. 1) were installed, one after another, at exit from the reactor; the temperatures of 280 K using a cryostat and 220 K using liquid nitrogen, respectively, were maintained in the heat exchangers. The condensed liquid was gathered in collector 10 (lined with fluoroplast-4), where its hydrogen fluoride and water content was measured by titration. The collector was also cooled with liquid nitrogen to a temperature of 220 K.

The gas flow was neutralized in alkaline absorber 11 after the collector. Samples for chromatographic and spectral analysis of gaseous reaction products were collected before and after the absorber into nickel tubes 12 and 13, respectively (the tubes were passivated with fluorine). Samples for gas analysis were taken from three to five times during one experiment.

Hydrogen and carbon monoxide, formed during high-temperature interaction of carbon and water vapor, were burned in ambient oxygen in device 14 (see Fig. 1).

After the experiment, the reactor was purged with an inert gas, the carbon material was cooled, mixed and a sample was taken. Methods for analyzing carbon and reaction products are described in [9].

### Experimental results and discussion

**Thermal state of the reactor.** The highest possible volume flow rate of oxygen was chosen for the experiments. The requirement imposed on the flow rate was that it would not lead to fluidization of the graphite layer. This flow rate value was  $90 \text{ cm}^3/\text{s}$ .

The change in the flow rate of the evaporated aqueous solution of hydrogen fluoride was in the range of  $5\text{--}85 \text{ mg/s}$ .

Measurements revealed that the graphite temperature in the layer near the exit of gas flows from the nozzle exceeded  $1400 \text{ K}$ , which is sufficient for the reaction producing water gas. A factor that had to be taken into account was that the nickel tubes where the thermocouples were installed distorted the temperature field due to their thermal characteristics (heat capacity and thermal conductivity); as a result, the measured temperature values were lower than the actual ones.

Fig. 3 shows the experimental temperature distributions recorded by thermocouples for two positions of the nozzle (see Fig. 2).

In position I, an asymmetric high-temperature zone whose shape was close to spherical formed in the reactor. The

asymmetry of the high-temperature zone is associated with interaction of forced and natural convection in the gas flow forming through the carbon layer. The gas flow formed by forced convection from the nozzle exit to the reactor exit; it was directed downwards. The gas flow formed by natural convection ( $T \approx 1300 \text{ K}$ ) moved from the nozzle exit upward, reached the cavity between the upper level of the carbon layer and the upper lid of the reactor ( $T \approx 400 \text{ K}$ ) and descended along the walls to the exit from the reactor.

The dependence of gas viscosity on temperature also affected the above-described structure of the flow: the viscosity of the gas increased with increasing temperature, and, consequently, the flow resistance of the gas increased in the high-temperature region, causing gas flows to bend around the high temperature region.

In position II, a symmetrical high-temperature region shaped as an ellipsoid stretched along the vertical axis was formed in the reactor. The components of forced and natural convection were collinear and directed towards the exit from the reactor.

The characteristic size of the zone with temperatures above  $1000 \text{ K}$  was approximately

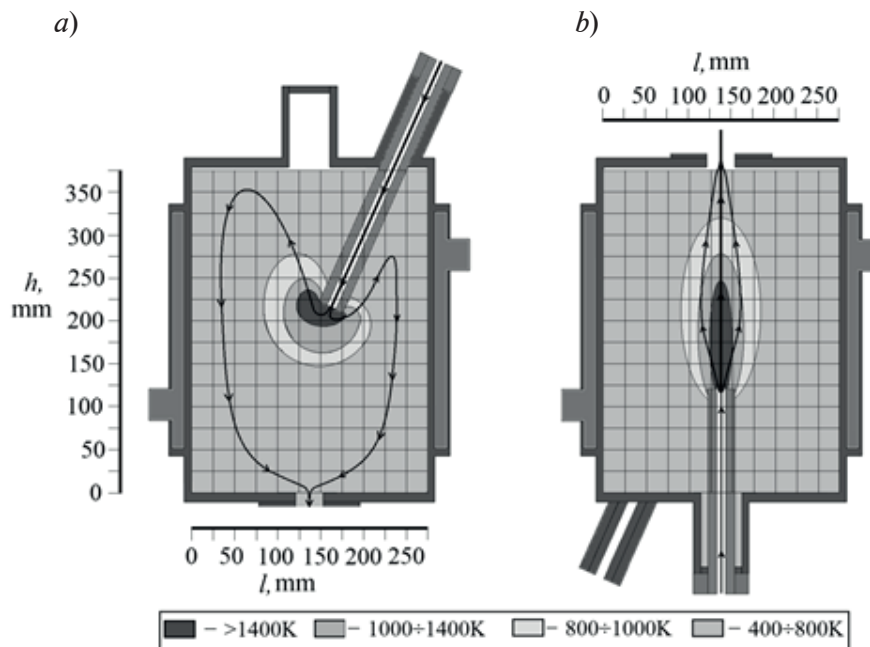


Fig. 3. Experimental temperature distribution in reactor for positions I (a) and II (b) of nozzle (see Fig. 2); projected flow paths of gas are also shown



7–8 cm in position I, and a region with characteristic heights of 13–15 cm and widths of 5–6 cm was formed in position II.

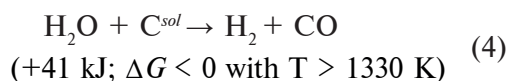
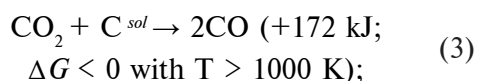
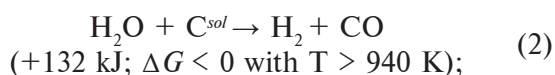
Water vapor stayed in the high-temperature ( $T > 1000$  K) zone for about 60% longer in position II than in position I with the same flow rate of gaseous components.

Three zones were observed in the high-temperature reaction region. The first was the oxygen zone that occupied the region from the point where the gas flowed from the nozzle to the surface where the oxygen concentration became zero. The second was the zone of interaction between water vapor and carbon that covered the region from the border of the oxygen zone to the surface where the temperature dropped to 940 K.

A highly exothermic reaction of interaction of carbon with oxygen occurred in the oxygen zone [11]. There was virtually no significant change in the water content in the oxygen zone as hydrogen formed by the water interacting with carbon was completely oxidized by oxygen.

We estimated the length of the oxygen zone in accordance with the recommendations given in [12]. The characteristic size of the oxygen zone for oxygen consumption of  $90 \text{ cm}^3/\text{s}$  and particle size distribution of carbon material given in Table 1 was 1.5 cm.

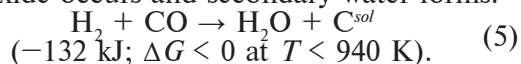
The oxygen zone is followed by the reduction zone where water vapor interacts with carbon, and hydrogen and carbon monoxide form at temperatures above 940 K. In addition, the following endothermic reactions can occur in this zone:



Here  $\Delta G$  is the variation of the Gibbs energy.

To find the region of temperature variation where the equilibrium in reactions (2)–(4) (as well as in reaction (5) below) is shifted to the right, we calculated the Gibbs energy variation depending on temperature.

The temperature is below 940 K in the region where secondary water forms. Under this condition, the reaction of interaction of water vapor with carbon stops but the reverse reaction of interaction of hydrogen with carbon monoxide occurs and secondary water forms:



Thus, the characteristic size of the oxygen zone is smaller than that of the high-temperature (above 1000 K) zone in the graphite layer by approximately an order of magnitude. It is preferable to maintain the temperature in the reduction zone in the range of 1000–1300 K, at which water vapor interacts with carbon, and reaction (4) is thermodynamically forbidden ( $\Delta G < 0$ ).

**Conversion of water vapor depending on the mass flow rate of the evaporated aqueous solution of hydrogen fluoride.** One of the parameters that determine the effectiveness of the proposed method for producing hydrogen fluoride is conversion of water vapor during high-temperature contact with carbon.

Conversion of water vapor at a given flow rate of aqueous solution of hydrogen fluoride was measured in two ways: by the composition of gaseous products at the exit of the reactor and by the amount of water gathered in collector 10 (see Fig. 1).

Measuring the composition of the gas phase at the exit from the reactor allows to calculate the conversion of water vapor based on the material balance of oxygen and hydrogen entering the reactor in the form of water vapor and molecular oxygen and leaving the reactor in the form of molecular hydrogen and carbon oxides.

Conversion of water vapor with respect to gaseous reaction products,  $K_{gas}$ , was calculated by the following formula:

$$K_{gas} = \frac{W_{\text{O}_2}}{W_{\text{H}_2\text{O}}} \times \frac{c_{\text{H}_2}}{\frac{c_{\text{CO}} - c_{\text{H}_2}}{2} + c_{\text{CO}_2} + c_{\text{O}_2}} \cdot 100\%, \quad (6)$$

where  $W$ , mg/s, is the flow rate of the corresponding components at the inlet of the reactor;  $c$ , vol.%, is the concentration of the corresponding substances in gas flow at the exit of the reactor after condensation of water and hydrogen fluoride.

Table 2 shows the typical composition of the gas phase after hydrogen fluoride condenser 10 (see Fig. 1).

The conversion of water vapor with respect to the liquid phase,  $K_{flu}$ , was determined as the ratio of the mass of water gathered in collector 10 (see Fig. 1) to the mass of water vapor fed to the reactor as part of aqueous solution of hydrogen fluoride.

Fig. 4 shows the values of water vapor conversion  $K_{gas}$  and  $K_{flu}$  depending on the flow rate of aqueous solution of hydrogen fluoride for two concentrations of hydrogen fluoride and two positions of the nozzle.



Table 2

**Characteristic composition of gaseous reaction products after hydrogen fluoride condenser**

Product	$c$ , vol.%	Detection method
CF <sub>4</sub>	–	Gas chromatography
CO	70	
CO <sub>2</sub>	20	
H <sub>2</sub>	10	
O <sub>2</sub>	0.5	
CH <sub>4</sub>	0.1	
(HF) <sub><math>n</math></sub>	0.1 with $n = 4$	Potentiometry
COF <sub>2</sub>	–	FTIR

Notes: accuracy for detecting the characteristic concentration  $c$  was  $10^{-2}$  vol.%; FTIR stands for Fourier-transform infrared spectroscopy

With the flow rate of aqueous solution of hydrogen fluoride varying from 5 to 90 mg/s, conversion of water vapor varies from 10 to 80–90%, passing through a maximum.

Decrease in water vapor conversion with an increasing solution flow rate from 20 to 90 mg/s is associated with a shorter period during which water vapor stays in the high-temperature zone, with the temperature of the carbon layer decreasing as it is cooled by flows of water vapor and hydrogen fluoride, and with the endothermic reaction of water gas formation.

The increase in conversion with the flow rate increasing from 5 to 20 mg/s is probably because the reaction products stay in the zone where secondary water forms for a shorter period of time.

The conversion of water vapor  $K_{gas}$  is higher than the conversion of  $K_{flu}$  in all experiments, by 14% on average. The likely reason for this is that finely dispersed water droplets are carried away from collector 10.

The depths of water vapor conversion for experiments with acid containing hydrogen fluoride at concentrations of 40% and 72% are close to each other. The conversion is significantly affected by the position of the nozzle. If the nozzle is installed axisymmetrically (in position II), the conversion of water vapor is higher by about 40%. This is likely because the flow stays in the high-temperature zone for a longer period.

#### Conversion of water vapor depending on the

**time it stays in the high-temperature zone.** The given time for which the flow stayed in the zone with temperatures above 1000 K was estimated approximately by the flow rate of aqueous solution of hydrogen fluoride and the configuration of the high-temperature zone, taking into account the oxygen flow. We assumed that the reaction forming water gas flows occurred outside the oxygen zone. The processed experimental data are given in Fig. 5.

The obtained dependences of the conversion depth on the time of contact of carbon with water vapor in the high-temperature oxygen-free zone allow to conclude that reaction (1) occurs almost completely with a contact time of about 7–10 s and this time does not depend on the initial concentration of hydrofluoric acid.

The obtained values of contact time required for 90% water vapor conversion are in good agreement with the data of [10], considering the interaction of water vapor with different grades of graphite in the absence of hydrogen fluoride. It was additionally established that the concentration of hydrogen fluoride in its initial solution does not affect the value of water vapor conversion.

Based on analysis of the data we have carried out, we can conclude that hydrogen fluoride has practically no effect on the rate of high-temperature interaction of water vapor with graphite at temperatures above 1000 K

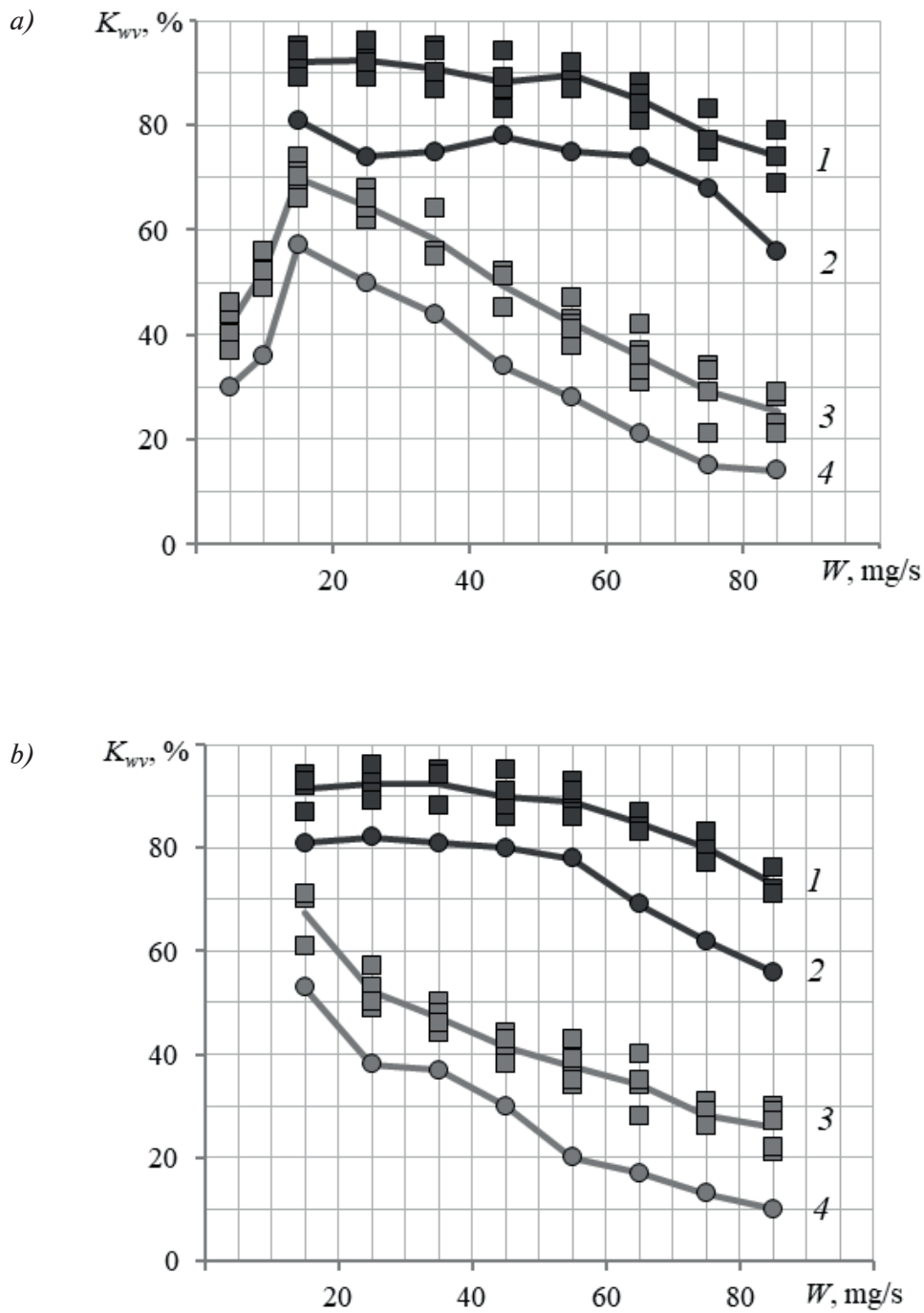


Fig. 4. Dependences for water vapor conversion:  $K_{wv}$ ,  $K_{gas}$  (curves 1, 3) and  $K_{flu}$  (2, 4) as functions of flow rate of aqueous HF solution upon contact of evaporated solution with high-temperature layer of carbon; HF content in solution was 40% (a) and 72% (b); Data are given for positions I (3, 4) and II (1, 2) of the nozzle (see Fig. 2)



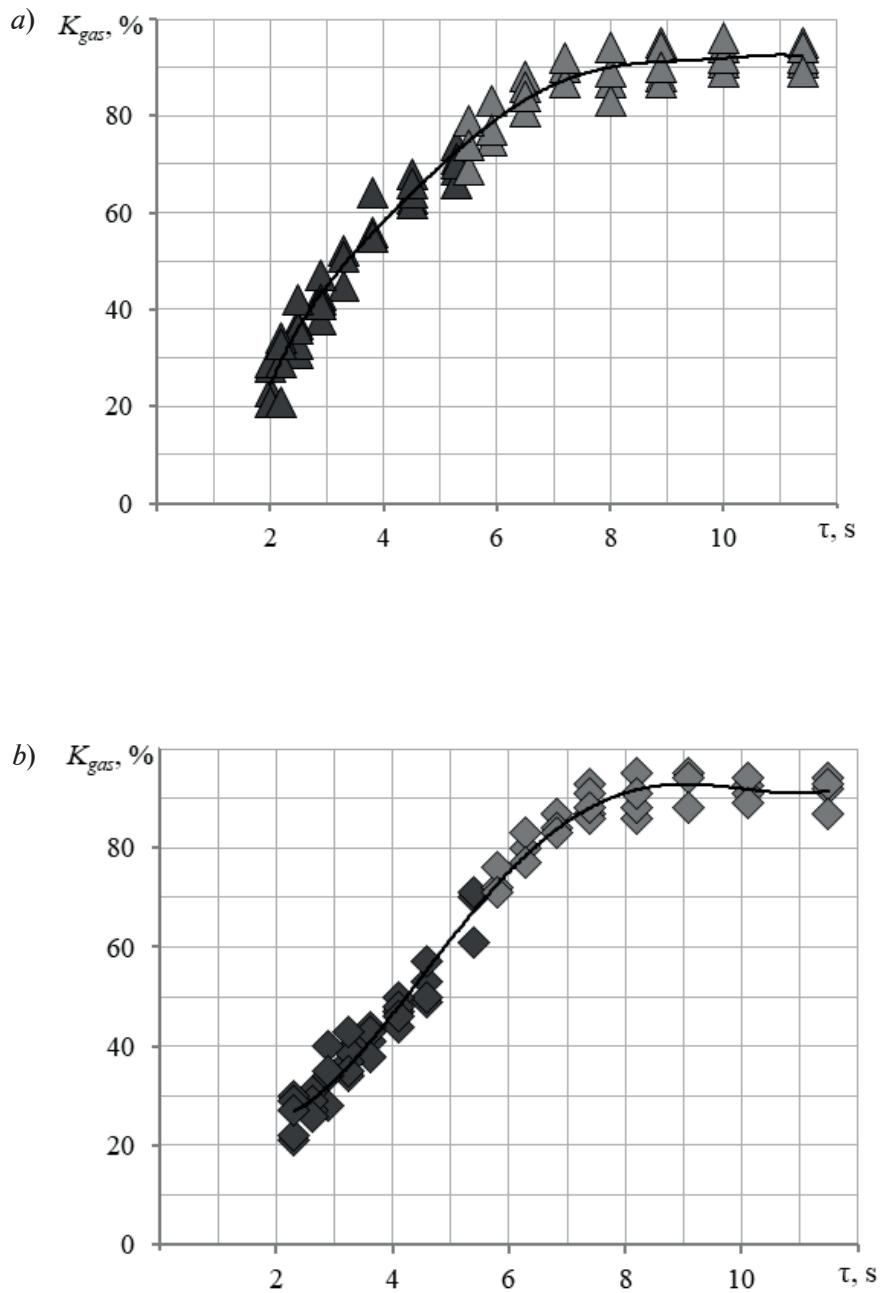


Fig. 5. Dependences  $K_{gas}$  for conversion of water vapor ( $wv$ ) with vaporized aqueous HF solution supplied to the high-temperature graphite layer as function of the time that  $wv$  stays outside the oxygen zone, in the layer with temperatures above 1000 K; HF content in solution was 40% (a) and 72% (b); data for positions I (black symbols) and II (light gray symbols) of nozzle are given (see Fig. 2)

### Conclusion

Based on the laboratory experiments that we have conducted, we have established the following.

The presence of hydrogen fluoride has almost no effect on the rate of reaction for interaction of water vapor with carbon at temperatures above 1000 K.

The characteristic time of contact of water

vapor with a fixed graphite layer at temperatures above 1000 K in the presence of hydrogen fluoride with 90% water vapor conversion is about 10 s.

The method of high-temperature interaction of water vapor with carbon can lay the groundwork for industrial technology for dehydration of aqueous solutions of hydrogen fluoride, including azeotropic.

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