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## Study of radiation-catalytic activity of aluminosilicate systems in the processes of hydrogen production from methane and methane-steam mixture

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The kinetics of H<sub>2</sub> accumulation during the radiation-thermocatalytic decomposition of CH<sub>4</sub>+ 2H<sub>2</sub>O and 2CH<sub>4</sub>+H<sub>2</sub>O mixtures at T=300÷773 K (P<sub>tot</sub>=1-12 atm) on the surface of aluminosilicate was studied. Doubling the relative concentration of methane in the reaction medium at T=773 K causes an increase in the yield of G(H<sub>2</sub>) from 1.2 to 2.3 molecule/100 eV. Based on the results obtained on the heterogeneous radiolysis of methane and the CH<sub>4</sub>+H<sub>2</sub>O mixture, the following conclusions can be drawn that the efficiency of energy transfer in the Al-Si + CH<sub>4</sub> system is less than in the Al-Si + H<sub>2</sub>O system, and the radiation-chemical yield of molecular hydrogen at heterogeneous radiolysis of methane in the presence of amorphous aluminosilicate at P<sub>CH<sub>4</sub></sub>≈1 atm, T=300÷773 K does not depend on temperature.

**Keywords:** γ-quanta, aluminosilicate, molecular hydrogen, methane, radiation-chemical yield.

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## Introduction

In order to identify effective ways to convert the energy of ionizing radiation into chemical energy using the universal energy carrier H<sub>2</sub>, this work investigated radiation-catalytic and radiation-thermocatalytic processes for the production of H<sub>2</sub> from methane and a mixture of CH<sub>4</sub>+H<sub>2</sub>O in the presence of amorphous aluminosilicate Al-Si.

As is known, the processes of catalytic conversion of methane are one of the main industrial ways of producing hydrogen and are characterized by energy intensity and multi-stage nature. In this case, part of the natural gas is used to supply energy to these processes, and part of the resulting hydrogen is used to restore nickel in the composition of the nickel-containing catalysts used. Therefore, research in the field of creating technological complexes for industrial processes for producing hydrogen with nuclear reactors is carried out in two directions:

- identifying the possibilities of using heat from high-temperature nuclear reactors to supply energy to the processes of producing hydrogen from methane;

- use of ionizing radiation energy obtained from nuclear fission to stimulate thermocatalytic processes for the production of H<sub>2</sub> [1-9].

In order to identify new effective catalysts for the conversion process of hydrogen production, various nanooxide catalysts, including aluminosilicate, are widely used [1-2, 10-13]. Therefore, studies of the laws of radiation-catalytic processes for producing hydrogen from methane and methane-steam mixtures are of great interest both in the field of atomic-hydrogen energy and in the field of selecting catalysts for thermocatalytic and radiation-thermocatalytic hydrogen production.

## I. Experimental technique

Amorphous aluminosilicate was taken as model and practically important objects of study. The catalyst

samples were preliminarily heat-treated in a flow of oxygen at  $T=923$  K for 24 hours. During heat treatment, the composition of the gases escaping in the oxygen flow was analyzed. The criterion for surface cleanliness was the absence of traces of CO and CO<sub>2</sub> in the composition of the exiting gas. Then the catalyst samples were transferred to a vacuum adsorption unit and subjected to heat treatment at  $T=873$  K for 8 hours. In the vacuum adsorption unit, in order to prevent oils and lubricants from getting on the catalyst samples, three special traps cooled with liquid nitrogen are built into the system. Thermal vacuum treatment of the samples continued at  $T=923$  K and  $P=10^{-3}$  Pa for 2 hours using only a zeolite pump, then the samples were cooled to room temperature while pumping with a zeolite pump. Adsorption of substances undergoing radiolysis was carried out from a graduated volume.

Chromatographic pure methane was used. Methane was preliminarily purified from moisture, CO and CO<sub>2</sub>, as well as impurities of other hydrocarbons by passing through adsorption columns, each 1 m long, with CaCl<sub>2</sub> sorbents, NaX molecular sieve and silica gel. Further purification was carried out in a vacuum adsorption unit by repeated freezing and pumping.

The samples were irradiated in sealed ampoules with  $\gamma$ -rays from a <sup>60</sup>Co isotope source. A theoretical calculation of the absorbed dose in heterogeneous systems was carried out [6] and methodological calculations of the absorbed dose of  $\gamma$ -radiation in heterogeneous systems were proposed. Our works [6-7] provide comparisons of the values of absorbed doses of  $\gamma$ -quanta in individual components of heterogeneous systems, determined by theoretical and experimental methods. It was established that the values of absorbed doses of  $\gamma$ -quanta in individual components of heterogeneous systems were determined by comparing the electron densities of the studied and dosimetric systems, and all theoretical calculated values coincide with each other within the accuracy of the determination [14-16]. Therefore, the absorbed dose rate of  $\gamma$ -quanta in the systems under study is determined based on the readings of chemical dosimeters - ferrosulfate, cyclohexane and methane according to the method [1, 6]:

$$D_i = \alpha \cdot D_{\text{dose}}$$

where,  $D_i$  is the absorbed dose rate in the systems under study,  $\alpha$  is the ratio of the electron densities of the studied and dosimetric systems, the values of which in our systems vary within the range  $\alpha=0.8\div0.9$ ;  $D_{\text{dose}}$  is the absorbed dose rate, determined by the chemical dosimetric method. The value of the absorbed radiation dose rate was determined as above.

## II. The discussion of the results

In early works [1], it was established that the maximum initial radiation-chemical yield of radiation-catalytic centers during heterogeneous radiolysis of water in optimal forms of catalysts is 7-8 particle/100 eV. For practical purposes, it is usually of interest to achieve relatively high yields of the energy carrier – hydrogen [1]. From the results obtained it is clear that heterogeneous radiolytic processes in the presence of catalysts - oxide

dielectrics, with an increased yield of products  $G(P)\geq 8$ , are exceeded only in the presence of chain channels for the transformation of intermediate products into final products.

Radiolysis and thermoradiolysis of pure methane were carried out under static conditions. The kinetics of hydrogen accumulation during heterogeneous radiolysis of methane in the presence of Al – Si was studied. In Fig. 1, the kinetic curves of H<sub>2</sub> accumulation during the heterogeneous radiolysis of methane in the presence of Al – Si are given, and on the basis of the kinetic curves the values of radiation-chemical yields  $G_{\text{tot}}(\text{H}_2)=0.75$  molecule/100 eV were determined. The change in CH<sub>4</sub> pressure in the reaction medium continues from 0.5 to 2 atm. The value of the radiation-chemical yield,  $G_{\text{tot}}(\text{H}_2)\sim 0.60\div0.75$  molecule/100 eV, does not cause any noticeable change. The values of the radiation-chemical yields of hydrogen, calculated for the energy absorbed by methane in these experiments, range from  $G_{\text{ads}}(\text{H}_2)\sim 20\div22$  molecule/100 eV, which exceeds the yield values for  $G_{\text{tot}}(\text{H}_2)$  radiolysis of CH<sub>4</sub> in the homogeneous state  $G_{\text{T}}(\text{H}_2)=5.7$  molecule/100 eV,  $G_{\text{ads}}(\text{H}_2)\geq G_{\text{T}}(\text{H}_2)$  [1, 6-7].

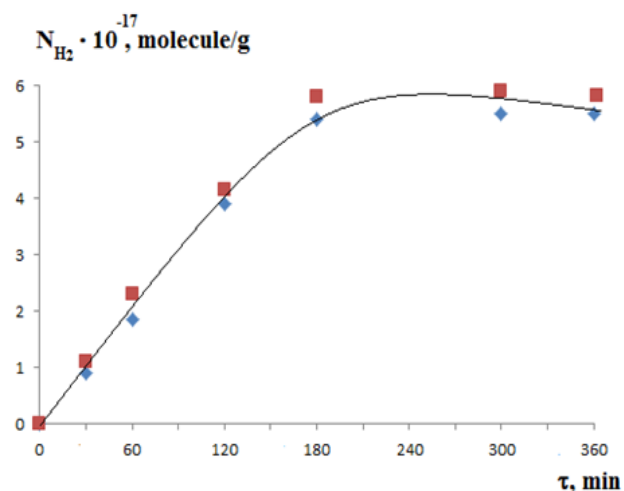


Fig. 1. Kinetics of accumulation of molecular hydrogen during radiolysis of CH<sub>4</sub> in the presence of Al-Si at  $T=300$  K,  $D_{\gamma}=1.1$  Gy/s

■ -  $P_{\text{CH}_4}=1$  atm., ♦ -  $P_{\text{CH}_4}=5$  atm.

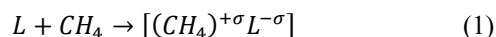
The observed high value of  $G_{\text{ads}}(\text{H}_2)$  indicates the presence of energy transfer from aluminosilicate to CH<sub>4</sub> molecules adsorbed on the Al-Si surface.

In order to influence the temperature on the yield of hydrogen during heterogeneous radiolysis of methane on the Al-Si surface, the kinetics of the accumulation of molecular hydrogen on the Al-Si surface at different temperatures was studied.

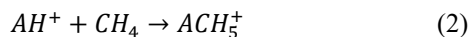
The effect of temperature on the yield of molecular hydrogen during heterogeneous radiolysis of CH<sub>4</sub> in the presence of Al-Si was studied. It has been established that the radiation-chemical yield of H<sub>2</sub> in the temperature range  $T=300\div773$  K, within the accuracy of determination, does not depend on temperature and  $G(\text{H}_2)\sim 0.75\div0.7$  molecule/100 eV.

As can be seen, the patterns of the influence of temperature on the values of radiation-chemical yields of

hydrogen during heterogeneous radiolysis of water and methane are the same (Table 1). Lewis and Brentsted acid sites can serve as active centers for the adsorption and radiation catalysis of CH<sub>4</sub> on the Al-Si surface. In the case of Lewis acid sites (L), adsorbed states are created on the surface:

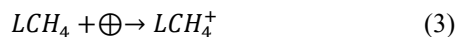


In the presence of Brentsted acid sites (AH<sup>+</sup>):

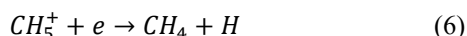
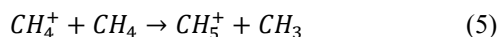


The lifetime of surface-adsorbed complexes depends on the strength of acid sites and ΔH processes (1) and (2). The energy of methane adsorption on the surface of aluminosilicate systems is less than the energy of water adsorption.

The strong adsorption bond of methane molecules on electron acceptor centers can cause significant deformations in the electronic structure of these molecules. As a result of this, one can expect a decrease in the ionization potential of methane molecules in adsorption complexes. In this state, methane molecules can interact with hot holes formed during the interaction of γ-quanta in the aluminosilicate system:



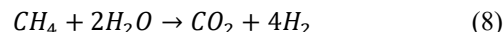
The resulting intermediate charged complexes can undergo both neutralization by electrons and ion-molecular transformation:



During the decay of methane molecules as a result of (5-7), the ratio between the yields of H<sub>2</sub> and nonequilibrium charge carriers G(H<sub>2</sub>)=½ G(n.c.c.) must be observed. At room temperature, due to the fact that methane is less adsorbed compared to water molecules, effective energy transfer in the Al-Si + CH<sub>4</sub> system does not occur and G(H<sub>2</sub>)< 1/2 G(n.c.c.).

During heterogeneous radiolysis of water at 300 K, in the presence of Al-Si, the majority of nonequilibrium charge carriers formed under the direct action of γ-quanta on Al-Si take part in the energy transfer process. In order to identify the possibility of additional channels for the

transformation of intermediate radical products of the decomposition of water molecules into molecular products as a result of interaction processes in the CH<sub>4</sub>+H<sub>2</sub>O system, heterogeneous radiolysis of the CH<sub>4</sub>+H<sub>2</sub>O mixture was studied. The conversion process of methane and water can be schematically represented by the equation:



The principles of heterogeneous radiolysis of a CH<sub>4</sub>+H<sub>2</sub>O mixture were studied at a stoichiometric ratio of components. The kinetics of radiation-catalytic, radiation-thermocatalytic and thermocatalytic accumulation of CO and H<sub>2</sub> was studied (Table 2).

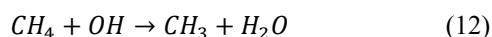
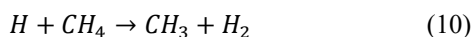
It has been established that in the composition of products, in addition to CO, CO<sub>2</sub> is formed in small quantities at high temperatures (T ≥ 773 K). At temperatures T ≥ 673 K, Al-Si - exhibits thermocatalytic activity during the transformation process in the CH<sub>4</sub>+H<sub>2</sub>O system. Therefore, in order to determine the kinetic parameters of the radiation component of radiation-thermocatalytic processes under identical conditions, the kinetics of accumulation of products during radiation-thermocatalytic and thermocatalytic decomposition of the CH<sub>4</sub>+H<sub>2</sub>O mixture was studied and from the difference W<sub>RT</sub> – W<sub>T</sub>, the rate of the radiation process was determined. In the temperature range 300÷373 K, the radiation-chemical yield of hydrogen does not depend on temperature and is equal to G<sub>total</sub>(H<sub>2</sub>)~0.40 molecule/100 eV. During heterogeneous radiolysis of the CH<sub>4</sub>+H<sub>2</sub>O system, energy transfer to the individual components of the system occurs only in the adsorption state. At low temperatures (T<373 K) in the heterogeneous system Al-Si+(CH<sub>4</sub>+2H<sub>2</sub>O) there is competition in the adsorption of individual components of the medium. Considering that water molecules are characterized by a relatively high heat of adsorption, at these temperatures the adsorption of water molecules will predominate on the Al-Si surface, which undergo radiation-chemical decomposition. Therefore, the radiation-chemical yield of molecular hydrogen corresponds to the limiting value of its yield during heterogeneous radiolysis of water in the presence of Al-Si at T=300 K. In the temperature dependence of the radiation-chemical yield in the coordinates lgG<sub>tot</sub>(H<sub>2</sub>) = f(1/T), more two regions T ~ 473÷773 K and T ≥ 773 K with activation energies of 2.1 and 46.70 kJ/mol. In these temperature ranges, both components of the CH<sub>4</sub>+2H<sub>2</sub>O system are in the gas base and the lifetime on the surface adsorption centers of these molecules is determined by the heat of adsorption (τ= τ<sub>0</sub>e<sup>Q/RT</sup>). On the surface, the process of capture of nonequilibrium charge carriers from water molecules will

Table 1.

Kinetic parameters of the process of hydrogen production during the radiation-catalytic decomposition of methane in the presence of amorphous aluminosilicate at P=1 atm., D=1,1 Gy/s

T, K	W <sub>T</sub> (H <sub>2</sub> ) molecule/g·s	W <sub>PT</sub> (H <sub>2</sub> ) molecule /g·s	G <sub>tot</sub> (H <sub>2</sub> ) molecule/100 eV	G <sub>ads</sub> (H <sub>2</sub> ) molecule/100 eV
300	-	-	0.75	22.00
673	2.67 · 10 <sup>14</sup>	6.67 · 10 <sup>14</sup>	0.75	25.01
773	3.33 · 10 <sup>14</sup>	8.01 · 10 <sup>14</sup>	0.84	29.12

prevail. This is also evidenced by the equality of hydrogen yields during thermoradiolysis of a mixture of  $\text{CH}_4 + 2\text{H}_2\text{O}$  and pure water in the presence of Al-Si at appropriate concentrations of water molecules in the reaction medium. In the temperature range 673-773 K, reactions of H and OH radicals with the original molecules can occur [17-18]:

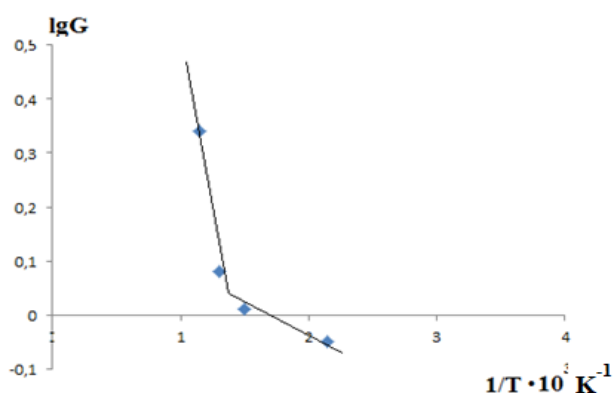


**Table 2.**

The influence of temperature on the value of the radiation-chemical yield of hydrogen during radiation-catalytic transformations of a mixture of  $\text{CH}_4 + 2\text{H}_2\text{O}$ , in the presence of an amorphous aluminosilicate at  $P_{\text{tot.}} = 1 \text{ atm.}$ ,  $D = 1,1 \text{ Gy/s}$

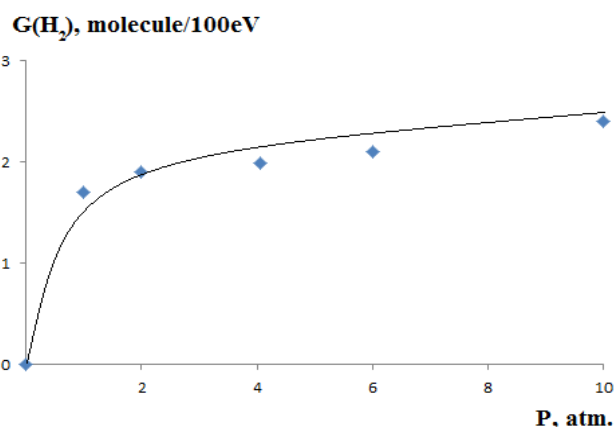
T, K	$G(\text{H}_2)$ , molecule/100eV	$G(\text{CO})$ , molecule/100 eV
300	0,40	-
373	0,40	-
473	0,90	-
673	1,05	-
773	1,20	0,45
873	2,70	0,90

As a result of these processes,  $G(\text{H}_2)$  doubles compared to the yield at  $T=373 \text{ K}$  and reaches a value of  $\sim 1 \text{ molecule/100 eV}$ . As can be seen from the fig. 2, with a further increase in temperature in the Al-Si+( $\text{CH}_4 + 2\text{H}_2\text{O}$ ) system, an energy-intensive stage of the process occurs, which leads to an increase in  $G(\text{H}_2)$  to  $2.7 \text{ molecule/100 eV}$ .



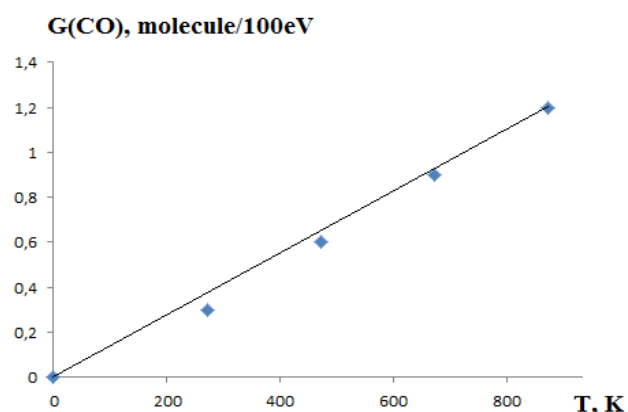
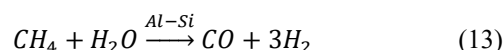
**Fig. 2.** Dependence  $\lg G = f(1/T)$  during thermoradiolysis of a  $\text{CH}_4 + 2\text{H}_2\text{O}$  mixture in the presence of Al-Si at  $P_{\text{tot.}} = 1 \text{ atm.}$  ( $D = 1,1 \text{ Gy/s}$ ).

The dependence of the radiation-chemical yield of molecular hydrogen, determined on the basis of kinetic curves, on the methane pressure in the reaction medium is presented in Fig. 3.



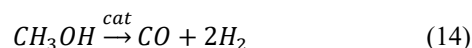
**Fig. 3.** Dependence of  $G(\text{H}_2)$  on  $\text{CH}_4$  pressure during heterogeneous radiolysis of methane in the presence of HLaY at  $T=300\text{K}$ ,  $D = 1,1 \text{ Gy/s}$ .

It is interesting to note that during heterogeneous thermoradiolysis of pure water in the presence of Al-Si, this region is not observed. At the same time, in the composition of the products of the process of heterogeneous radiolysis of the  $\text{CH}_4 + 2\text{H}_2\text{O}$  mixture, CO is observed, which was absent before this region (Fig.4). The activation energy for CO accumulation in this temperature range, within the accuracy of determination, coincides with the activation energy for  $\text{H}_2$  accumulation ( $47.1 \text{ kJ/mol}$ ). The radiation-chemical yield of CO at  $T=873 \text{ K}$  is equal to  $G(\text{CO})=0.90 \text{ molecule/100 eV}$  and at the same time the ratio  $G_{\text{total}}(\text{H}_2)/G(\text{CO}) \sim 3$ . No  $\text{CO}_2$  is observed in the composition of the process products, which indicates that radiation-thermal catalytic decomposition in the Al-Si+( $\text{CH}_4 + 2\text{H}_2\text{O}$ ) system occurs not according to (8), but according to the equation:



**Fig.4.** Dependence of radiation-chemical yield  $G(\text{CO})$  on temperature.

The formation of CO apparently occurs as a result of the thermocatalytic decomposition of  $\text{CH}_3\text{OH}$ , formed according to (11) in the region  $T \geq 773 \text{ K}$ :



The yield of OH radicals that escaped recombination with H atoms is equal to the yield of H atoms that interact with the original molecules according to (9-10). A more

probable process of consumption of these OH radicals on the Al–Si surface is reaction (11). The decomposition of the formed CH<sub>3</sub>OH according to (13) should give an increase in G(H<sub>2</sub>) ~ 2 molecule/100 eV, which is observed experimentally at T~873 K. The rates of reactions (10-11) under identical experimental conditions depend on the concentration of methane in the reaction medium. The kinetics of H<sub>2</sub> and CO accumulation during the radiation-thermocatalytic decomposition of CH<sub>4</sub>+2H<sub>2</sub>O and 2CH<sub>4</sub>+H<sub>2</sub>O mixtures at T=773 K (P<sub>tot</sub>=1 -5 atm) was studied. An increase in the relative concentration of methane in the reaction medium by a factor of two at T=773 K causes an increase in the yield of G(CO) from ~ 0.9 to 1.81, and G(H<sub>2</sub>) from 1.2 to 2.3 molecule/100 eV.

## Conclusion

Thus, based on the results obtained on the heterogeneous radiolysis of methane and the CH<sub>4</sub>+H<sub>2</sub>O mixture, the following conclusions can be drawn:

- the efficiency of energy transfer in the Al–Si+CH<sub>4</sub> system is less than in the Al–Si+H<sub>2</sub>O system; the radiation-chemical yield of molecular hydrogen during heterogeneous radiolysis of methane in the presence of amorphous aluminosilicate at R<sub>CH<sub>4</sub></sub>~1 atm, T=300÷773 K

does not depend on temperature;

- during heterogeneous radiolysis of a CH<sub>4</sub>+H<sub>2</sub>O mixture, there is competition between the processes of interaction of the components of the mixture with the active centers of the Al–Si surface; in this competition, one of the determining factors is the energy of adsorption interaction with surface active centers; the Al–Si +(CH<sub>4</sub>+H<sub>2</sub>O) system is dominated by the radiation-catalytic decomposition of water and the release of H<sub>2</sub>, which corresponds to the yield of heterogeneous radiolysis of water.

- during heterogeneous radiolysis of a mixture of CH<sub>4</sub>+H<sub>2</sub>O, at T ≥ 673 K, the transformation of intermediate products of the decomposition of water H and OH into molecular products H<sub>2</sub> and CO occurs as a result of their interaction with CH<sub>4</sub> molecules. Radiation-thermocatalytic steam conversion of methane in the presence of amorphous aluminosilicate occurs in the temperature range T ≥ 773 K.

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## Дослідження радіаційно-каталітичної активності алюмосилікатних систем у процесах одержання водню з метану та метано-водяної суміші

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Вивчено кінетику накопичення  $H_2$  під час радіаційно-термокatalітичного розкладу сумішей  $CH_4 + 2H_2O$  та  $2CH_4 + H_2O$  при  $T = 300-773$  К ( $P_{tot} = 1-12$  атм) на поверхні алюмосилікату. Подвоєння відносної концентрації метану в реакційному середовищі при  $T = 773$  К спричиняє зростання виходу  $G(H_2)$  від 1,2 до 2,3 молекули/100 еВ. На основі отриманих результатів з вивчення гетерогенного радіолізу метану та суміші  $CH_4 + H_2O$  можна зробити висновок, що ефективність передачі енергії в системі  $Al-Si + CH_4$  є меншою, ніж у системі  $Al-Si + H_2O$ . Радіаційно-хімічний вихід молекулярного водню при гетерогенному радіолізі метану в присутності аморфного алюмосилікату за умов  $PCH_4 \approx 1$  атм,  $T = 300-773$  К не залежить від температури.

**Ключові слова:**  $\gamma$ -кванти, алюмосилікат, молекулярний водень, метан, радіаційно-хімічний вихід.