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DEPENDENCE OF THE RATE OF CORROSION AND HYDROGEN DIFFUSION OF 09MN2SI STEEL ON THE CONCENTRATION OF HYDROGEN SULPHIDE IN CHLORIDE-ACETATE ENVIRONMENTS

Bohdan Datsko; Maryan Chuchman; Vasyl Ivashkiv; Svitlana Halaichak

Karpenko Physico-Mechanical Institute of National Academy
of Science of Ukraine, Lviv, Ukraine

Summary. *It is found that with increasing concentration of hydrogen sulphide (H_2S) to 100, 1000 and 2800 mg/dm³ (H_2S_{sat}) the corrosion rate (C) of steel 09Mn2Si increases by ~1,48; 1,58 and ~1,64 times in 24 hours of exposure, however, in 720 h, it increases by ~1,8 and ~3,3 times at its concentration of 1000 mg/dm³ and saturation, while at 100 mg/dm³ C decreases by 1,8 times, which is due to the formation of continuous sulphide films. It is shown that the volume amount of hydrogen in 09Mn2Si steel increases with the increase of H_2S content of the solution from 100; 500; 1500 and 2800 mg/dm³ in 1,2; 1,5; 1,9 and 2,5 times. Hydrogen diffusion increases from $0.9 \cdot 10^{-6}$ to $2.7 \cdot 10^{-6}$ cm²/s with the increase of membrane thickness from 0,75 to 1,50 mm and does not depend on the H_2S content.*
Key words: *corrosion, hydrogenation, hydrogen sulfide, hydrogen, diffusion.*

Statement of the problem. It is known that hydrogen sulfide corrosion and hydrogen embrittlement are one of the main causes of the destruction of various equipment and pipelines in the oil and gas production and processing industries [1–4]. In connection with the active development of the Black Sea shelf with national reserves of oil and gas, the need for systematized data on the anti-corrosion protection of hydraulic structures in the presence of hydrogen sulfide in seawater, which is very corrosive, is growing. At present, there are no established theoretical ideas about the impact of different concentrations of hydrogen sulfide on the specified corrosion processes, and the previously proposed approaches for determining the susceptibility of steels to hydrogen sulfide corrosion cracking are those that require significant improvement.

Analysis of available investigation results. Dissolved hydrogen sulfide, which is contained in mining products, increases the rate of corrosion of steels and contributes to their intensive flooding [5–8]. It is believed [6] that one of the main factors of corrosion-mechanical destruction is hydrogen embrittlement of metals, which is directly related to the occurrence of hydrogen depolarization during their corrosion. The influence of hydrogen sulfide media of different chemical composition on the corrosion-mechanical properties and corrosion of steels and alloys is described in detail in the literature. However, little attention has been paid to the effect of hydrogen penetration through the metal surface of metals [9–11]. Diffusion of hydrogen atoms into the crystal lattice of the metal and its continuous accumulation in close to surface layers with increased defects results in its molization or the formation of hydride phases. This creates additional internal stresses and results in the formation of cracks and accelerated destruction of the surface layers of the metal. Practically all metals are characterized by increased destruction under hydrogen influence, but iron-carbon alloys and hydride-forming metals are the most sensitive. In the first case, molalized hydrogen in structural defects generates internal stresses, which facilitates the destruction of surface layers [12]. Hydride

transformation in the surface layers of metals significantly affects the processes of destruction, accelerating or reducing the rate of destruction of surfaces [13, 14].

The objective of the paper. The objective of the paper is to determine the relationship between the rate of corrosion, flooding and diffusion of hydrogen through membranes of different thicknesses made of structural 09Mn2Si steel at different concentrations of hydrogen sulfide.

The results of the investigation. 09Mn2Si steel is investigated, the chemical composition is given in the table. 1. The following media are used for the investigation: 5% aqueous solution of NaCl + 0,5% CH$_3$COOH, without (pH = 4. 22±3ºC) and with different concentrations of H$_2$S (pH = 2.7 – 4. 22±3ºC). Most of the investigations on the influence of hydrogen sulfide on the rate of corrosion and corrosion of steels are carried out in chloride-acetate media saturated with it, but the content of hydrogen sulfide in Ukrainian deposits is much lower. Therefore, the effect of its concentration on corrosion on hydrogen diffusion is investigated. Hydrogen sulfide is obtained by hydrolysis of aluminum sulfide, which guaranteed its purity, and its solutions were prepared by passing the gas mixture of hydrogen sulfide and argon of appropriate concentrations through the working solution. The concentration of hydrogen sulfide in the solution is determined by the iodometric method according to GOST 22387.2-97. In order to prepare the medium, reagents of «h.c.» and «c.d.a.» qualification are used. The value of hydrogen index of the working solutions is controlled by pH-meter pH-673.M with glass electrode ESL-6307.

Table 1

Chemical composition of the investigated materials

Material	Content of elements, % by mass							
	C	Si	Mn	Ni	Cr	Cu	S	P
09Mn2Si	≤ 0.12	$0.5 - 0.8$	$1.3 - 1.7$	≤ 0.3	≤ 0.3	≤ 0.25	≤ 0.035	≤ 0.03

Electrochemical investigations are carried out using IPC-Pro potentiostat and three-electrode cell. The potentials are determined relatively to the saturated silver chloride electrode reference (E=0.203 V), the auxiliary electrode is platinum, the speed of the potential sweep is 1.0 mV/s. The investigations are carried out on the samples 15×10×2 mm in size. Their operating part is sandpapered by P1200 sandpaper.

Corrosion rate is investigated by the gravimetric method according to ISO 7384:1986 [15], on samples 20×30×2 mm in size.

Diffusion of hydrogen through membranes of different thicknesses made of 09Mn2Si steel is determined by the electrochemical Devanathan–Stakhurskyi method [9], using double electrochemical cell (Fig. 1) on samples with thickness 0.75; 1.0 and 1.5 mm, with operating area in the cell 1.0 sm^2. Before the investigation the samples are degreased with ethanol, then the cell is mounted, dividing it into two parts: the input (cathode), into which, after reaching the equilibrium current density, 5% NaCl + 0.5% CH$_3$COOH solution with different concentrations of hydrogen sulfide is poured, and the output (anode), where the surface is polarized +200 mV in a 0.1 N solution of NaOH + 0.1 N Na$_2$MoO$_4$, and the transition of anodic current to steady value J_i^∞ is recorded, and converted into hydrogen penetration current according to the following equation [10]:

$$J_\infty = I_\infty / FA \qquad (1)$$

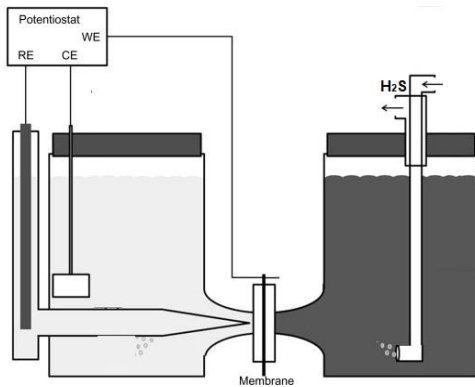where *A* is the area of the sample through which diffusion occurs, and *F* is Faraday constant.

Figure 1. Schematic representation of electrochemical cell for determining hydrogen permeability
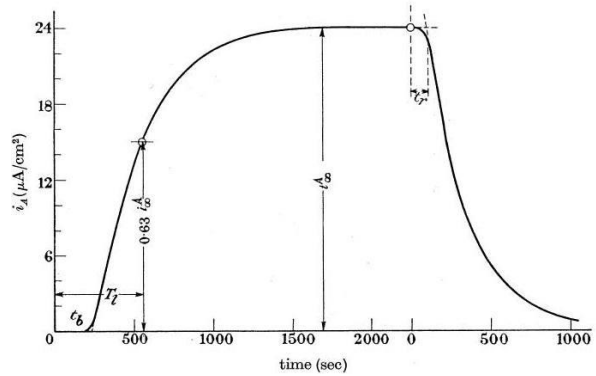

Figure 2. Schematic representation of the dependence of the anodic current on time

The effective diffusivity of hydrogen D_{eff} is calculated according to the equation:

$$D_{eff} = d^2/6\ t_L \qquad (2)$$

where d is the membrane thickness and t_L is the time in seconds for $0.63\ J_i^\infty$ saturation, which is extrapolated from the penetration curve [Fig. 2].

The concentration of hydrogen on C_0 cathode side can be estimated by the following formula [16]:

$$C_0 = J_\infty d / D_{eff} \qquad (3)$$

On the basis of electrochemical investigations at the beginning of the exposure, it is determmined that the saturation of chloride-acetate solution with hydrogen sulfide results in the shift of the potential of 09Mn2Si steel in more negative direction by ~60 mV. It is shown that there is no plateau on the cathodic curves (Fig. 3), which is characteristic for hydrogen depolarization. Since the angle of inclination of the cathodic curves is greater than that of the anodic curves, the rate of corrosion is determined by the cathodic process. It should be noted that the corrosion rate of 09Mn2Si steel in the solution of 5% NaCl+0.5% CH₃COOH is ~4 times lower than that of NACE (Table 2).
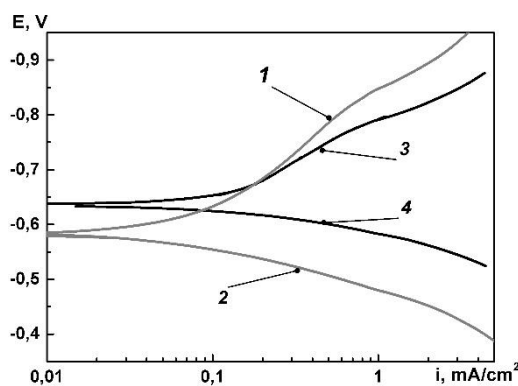

Figure 3. Polarization curves of 09Mn2Si steel in solution of 5% NaCl + 0.5% CH₃COOH: *1, 2* – without H₂S; *3, 4* – with H₂S
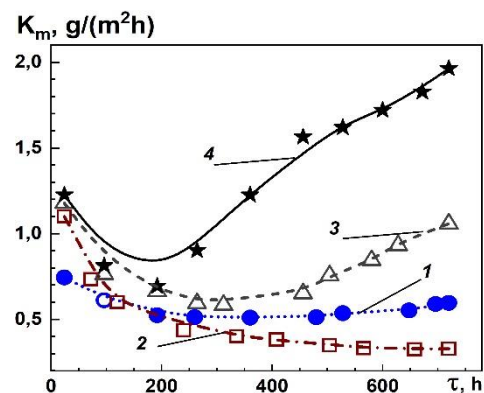

Figure 4. Corrosion rate of 09Mn2Si steel in chloride-acetate media with different concentrations of H₂S: 1 – without H₂S; 2 – 100 mg/dm³; 3 – 1000 mg/dm³; 4 – saturated (2800 mg/dm³)

Table 2

Electrode potentials and corrosion rate of 09Mn2Si steel in chloride-acetate media

Environment	E, mV	i, mA/SM^2	g, g/(m^2·h)	h, mm/year
5% NaCl+0,5% CH$_3$COOH	-0.580	0.030253	0.3161	0.3518
NACE	-0.642	0.116607	1.2183	1.3561

The gravimetric method shows that the corrosion rate of 09Mn2Si steel in the solution of 5% NaCl + 0.5% CH$_3$COOH without hydrogen sulfide during exposure up to 720 h (Fig. 4 curve *1*) is within 0.51...0.74 g/(m^2·h), by 480 h it decreases by 1.5 times to 0.51 g/(м^2·h), and then gradually increases by ~1.2 times to ~0.60 g/(м^2·h). According to the saturation of the solution with hydrogen sulfide of 100 mg/dm^3 (Fig. 4, curve *2*), it gradually decreases from ~1.1 to ~0.33 g/(м^2·h) for exposures up to 720 h, and is greater than in the medium without hydrogen sulfide only for exposure up to 120 h in ~1.5 ... 1.1 times, further it is smaller ~1.1 ... 1.8 times due to the formation of passivating films (troilite).

In the solution with hydrogen sulfide concentration of 1000 mg/ dm^3 for 720 h of the investigation, the corrosion rate is within ~1.18...0.58 g/(м^2·h)m which is higher on average than in the solution without and with H$_2$S (100 mg/ dm^3) in ~1.5...1.1 times and ~1.1...1.8 times. During exposure from 24 to 300 h, it decreases by ~2.0 times up to 0~.58 g/(m^2·h) (Fig. 3.2 curve *3*), with further exposure to 720 h, it increases to ~1.1 g/ (m^2·h), which indicates the formation of more loose and corrosion-resistant sulfide films (mackinavite + kansite).

Upon saturation of 5% NaCl + 0.5% CH$_3$COOH solution with hydrogen sulfide (Fig. 4, curve *4*), the corrosion rate decreases from the beginning of exposure to 192 h by ~1.8 times from ~1.2 to 0.69 g/(m^2·h), respectively, by 720 h of exposure gradually increases to ~1.96 g/(m^2·h), which is ~1.6 times more than at the beginning of the investigation. In general, during 720 hours, it is within the range of ~1.96...0.69 g/(m^2·h) and is greater on average by ~3.6...1.4; 1.18...2.1 and 1.6...1.2 times than in chloride-acetate solution without and with hydrogen sulfide concentration of 100 and 1000 mg/ dm^3.

The nature of the change in anodic current curves after reaching the maximum, namely its decrease with increasing concentration of hydrogen sulfide in the chloride-acetate medium, is due to the formation of sulfide films on the membrane surface in the input part of the cell (Fig. 5). The increase in the maximum anodic current with the increase in the thickness of the membrane occurs due to the uneven distribution of cathodic and anodic areas of corrosion on the input membrane.

From the obtained time dependences of the anodic current for hydrogen sulfide concentrations of 100, 500, 1500 mg/dm^3 in chloride-acetate medium, it is determined that with the increase in the membrane thickness from 0.75 mm to 1.5 mm, the effective passage time of hydrogen through the membrane increases in all media , which is caused by the duration of passage of hydrogen through the membrane (Fig. 5, Table 3). The increase in the maximum anodic current in 09Mn2Si steel due to the increase in the membrane thickness occurs due to the uneven distribution of cathode and anode areas on the input side of the membrane.
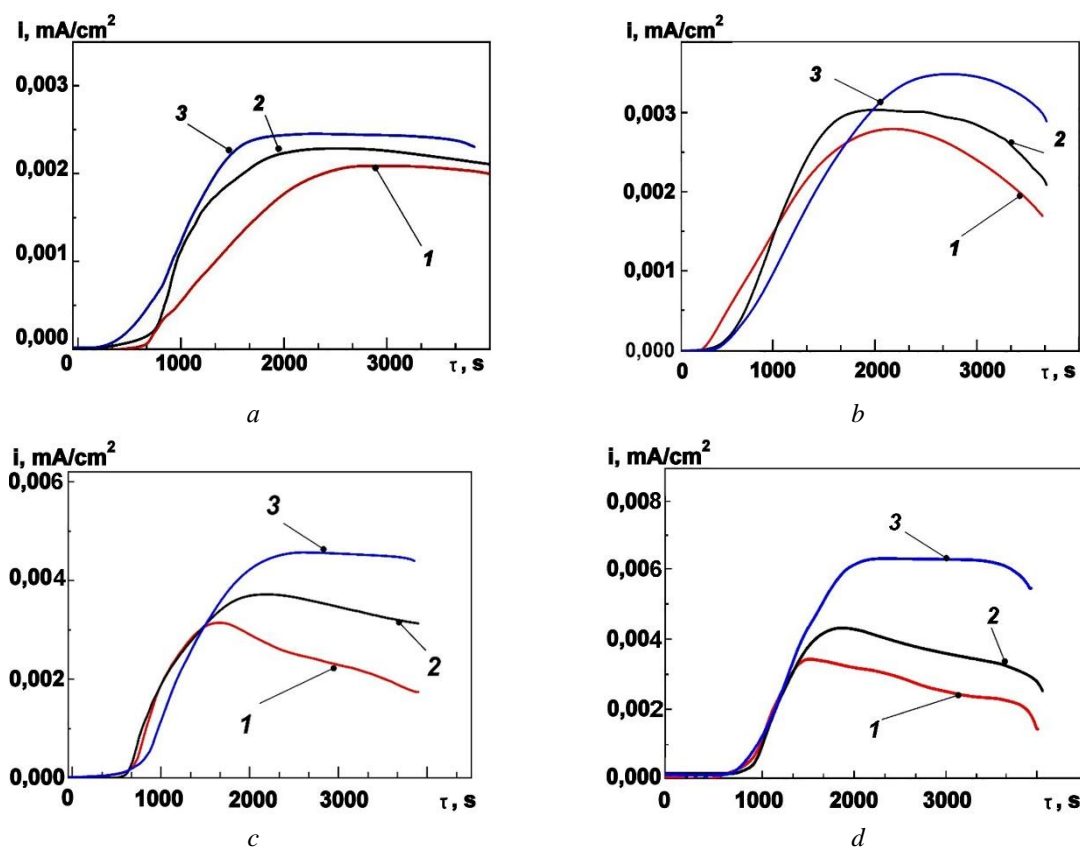
Figure 5. Change in the anodic current of membranes of different thicknesses in chloride-acetate medium with different concentrations of H$_2$S: 100 mg/dm^3 (a); 500 mg/dm^3 (b);
1500 mg/dm3 (c); 2800 mg/dm^3 (d): 1 – 0.75 mm; 2 – 1.0 mm; 3 – 1.5 mm

Table 3

Hydrogen permeability parameters calculated from the time dependences of the anode current

C_{H2S}, mg/dm^3	L, cm	i, mA/cm^2	t, c	$i_{0,63}$, mA/cm^2	$D_0 \cdot 10^{-7}$, cm^{2}c^{-1}	$J \cdot 10^{-11}$, mol·cm^{-2}c^{-1}	$C_0 \cdot 10^{-6}$, mol·cm^{-3}
2800	0.075	0.0034	1156	0.0021	8.2	3.5	3.6
	0.100	0.0044	1272	0.0028	13.0	4.6	3.5
	0.150	0.0064	1380	0.0040	27.0	6.6	3.7
1500	0.075	0.0031	1020	0.0020	9.2	3.2	2.6
	0.100	0.0037	1148	0.0023	15.0	3.8	2.6
	0.150	0.0045	1410	0.0028	27.0	4.7	2.6
500	0.075	0.0028	1044	0.0018	8.9	2.9	2.4
	0.100	0.0031	1072	0.0019	16.0	3.2	2.0
	0.150	0.0035	1432	0.0021	26.0	3.6	2.1
100	0.075	0.0023	1048	0.0014	8.9	2.3	1.9
	0.100	0.0025	1020	0.0015	16.0	2.5	1.6
	0.150	0.0022	1560	0.0014	24.0	2.2	1.6

The value of effective diffusion coefficient of 09Mn2Si steel, which is within the range of 8.2 10^{-7}–8.9 10^{-7}cm^{2}c^{-1} for 0.75 mm membrane thickness in chloride-acetate medium with increasing hydrogen sulfide concentration from 100 to 2800 mg/dm^3, for 0.10 mm thickness –

$1.3 \cdot 10^{-7}$–$1.6 \cdot 10^{-7}$ cm^{2}c^{-1} and for 0.15–2.4 10^{-7}–2.7 10^{-7}cm^{2}c^{-1}, respectively is determined. This thickness distribution is also related to the uneven distribution of cathodic and anodic areas on the membrane input side due to corrosion in chloride-acetate medium with different concentrations of hydrogen sulfide, which results in the increase of anodic currents, in contrast to the uniform cathodic polarization of the membrane surface for traditional hydrogen diffusion measurement by Devanathan-Stahurskyi method.
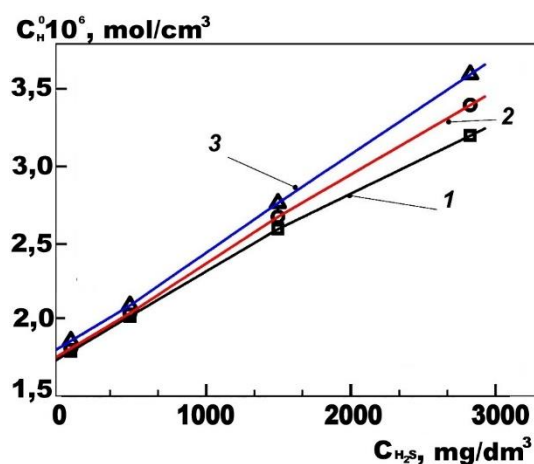

Figure 6. Dependence of the concentration of bulk hydrogen in the metal of membranes of different thicknesses in chloride-acetate medium on the content of hydrogen sulfide: 1 – 0.75 mm; 2 – 1.0 mm; 3 – 1.5 mm

It is determined that the volume of hydrogen in the metal, based on the electrochemical method of hydrogen diffusion through membranes with different thicknesses, increases linearly with the increase in the concentration of hydrogen sulfide in the medium, regardless to the membrane thickness from $1.6 \cdot 10^{-6}$ to $3.7 \cdot 10^{-6}$ mol·cm^{-3}, that is in 1.3; 1.5 and 2.1 times (Fig. 6).

Conclusions. On the basis of electrochemical investigations it is determined that when chloride-acetate solution is saturated with hydrogen sulfide at the beginning of exposure for 1 hour, the corrosion rate of 09Mn2Si steel increases by ~4.0 times and is controlled by the cathodic process. According to long-term investigations using gravimetric method, it is shown that the corrosion rate increases by 1.8 and 3.3 times at hydrogen sulfide concentration 1000 mg/dm^3 and saturation (2800 mg/dm^3) from 0.59 to 1.06 and 1.96 g/(m^2·h), on the other hand for 100 mg/dm^3 it increases by 1.8 times to 0.33 g/(m^2·h) in comparison with chloride-acetate solution without hydrogen sulfide, which is due to the formation of solid, non-loose sulfide films.

The investigation of hydrogen diffusion through membranes of different thicknesses made of 09Mn2Si steel using the electrochemical method of Davanathan-Stakhurskyi showed that the volumetric amount of hydrogen increases linearly with the increase in the concentration of hydrogen sulfide in chloride-acetate solution from 100; 500; 1500 та 2800 mg/dm^3 by 1.2; 1.5; 1.9 and 2.5 times from $1.4 \cdot 10^{-6}$ to $1.6 \cdot 10^{-6}$; $2.2 \cdot 10^{-6}$; 2.6 and 3.5 10^{-6} mol/cm^3 respectively.

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ЗАЛЕЖНІСТЬ ШВИДКОСТІ КОРОЗІЇ ТА ДИФУЗІЇ ВОДНЮ СТАЛІ 09Г2С ВІД КОНЦЕНТРАЦІЇ СІРКОВОДНЮ В ХЛОРИДАЦЕТАТНИХ СЕРЕДОВИЩАХ

Богдан Дацко; Мар'ян Чучман; Василь Івашків; Світлана Галайчак

Фізико-механічний інститут імені Г. В. Карпенка Національної академії наук України, Львів, Україна

Резюме. *Досліджено швидкість корозії електрохімічним і гравіметричним методами та дифузію водню методом Деванатхана-Стахурського сталі 09Г2С у хлоридно-ацетатних середовищах з різною концентрацією сірководню. Встановлено, що зі зростанням концентрації сірководню у хлорид-ацетатному середовищі до ~100, 1000 мг/дм³ та ~2800 мг/дм³ швидкість корозії сталі 09Г2С зростає в ~1,48; 1,58 та ~1,64 раза за 24 год експозиції. За 720 год вона збільшується в ~1,8 та ~3,3 раза за його концентрації ~1000 мг/дм³ та насиченням відповідно, натомість за ~100 мг/дм³ вона знижується в ~1,8 раза. Це зумовлено формуванням суцільних сульфідних плівок на поверхні сталі. Електрохімічними дослідженнями показано, що за насичення сірководнем розчину 5% NaCl + 0,5% CH₃COOH на початку експозиції за 1 год швидкість корозії сталі 09Г2С зростає у ~4,0 раза та контролюється катодним процесом. Збільшення концентрації сірководню від 0 до 1,5 г/дм³ супроводжується зміщенням потенціалів корозії в область від'ємніших значень: від -0,58 до -0,64 В і зростанням швидкості корозії від 0,072 до 0,172 мА/см². У всіх випадках корозія зразків перетікає за катодного контролю. Показано, що об'ємна кількість водню в сталі 09Г2С зростає лінійно зі збільшенням вмісту H₂S розчини від 100; 500; 1500 та 2800 мг/дм³ в 1,2; 1,5; 1,9 та 2,5 раза відповідно та не залежить від товщини мембрани. Дифузія водню натомість підвищується від 0,9·10⁻⁶ до 2,7·10⁻⁶ см²/с зі зростанням товщини від 0,75 до 1,50 мм і не залежить від вмісту H₂S, а незначний її розподіл за товщиною мембрани 8,2·10⁻⁷ - 8,9·10⁻⁷, 1,3·10⁻⁷ - 1,6·10⁻⁷ та 2,4·10⁻⁷ - 2,7·10⁻⁷ см²с⁻¹ пов'язаний зі нерівномірним розподілом катодних та анодних ділянок на вхідній стороні мембрани внаслідок утворення сульфідних плівок на поверхні мембрани, що призводить до зростання максимуму анодного струмів та подальшу зміну характеру анодних кривих, а саме, їх зменшення з часом та зростання з підвищенням концентрації сірководню у розчині на відміну за рівномірної катодної поляризації поверхні мембрани для традиційного вимірювання дифузії водню методом Деванатхана-Стахурського.*

Ключові слова: *корозія, наводнювання, сірководень, водень, дифузія.*