

S. V. Frolenkova¹, Cand. Sc. (Tech.),
orcid.org/0000-0002-6727-2903,
V. I. Vorobyova¹, Cand. Sc. (Tech.),
orcid.org/0000-0001-7479-9140,
T. A. Overchenko¹, Cand. Sc. (Tech.),
orcid.org/0000-0002-5883-6228,
M. I. Skiba², Cand. Sc. (Tech.),
orcid.org/0000-0001-9686-736X

1 – National Technical University of Ukraine “Igor Sikorsky Kyiv Polytechnic Institute”, Kyiv, Ukraine, e-mail: svetlana.frolenkova@gmail.com

2 – Ukrainian State University of Chemical Technology, Dni-pro, Ukraine, e-mail: Margaritaskiba@gmail.com

ECOLOGICALLY SAFE MODIFIED SOLUTIONS FOR DEPOSITION OF CONVERSION COATINGS ON STEEL

Purpose. To analyze the impact of the nature of solution components on the process of conversion coating formation on low-carbon steel.

Methodology. Coatings were obtained by exposing samples to the modifying solution at an ambient temperature for certain time. The potentiostatic and potentiodynamic polarization were employed to investigate solution components' impact on the steel surface behavior. Conversion film content was determined with an X-ray microscope. Pictures of the obtained coatings were derived with optical and electron microscopes. The obtained conversion coatings corrosion protection efficiency was determined by the gravimetric measurements.

Findings. A number of cold solutions for the formation of modified oxide conversion coatings on low-carbon steel for temporary protection against corrosion in liquid and air environments have been elaborated. As a basis for such mixtures, zinc acetate solutions are proposed. As modifying additives oxoanions were used whose incorporation into oxide conversion films was confirmed by X-ray scanning microscopy. It was determined that the holding time increase leads to a rise in content of modifying components in the film and growth of its thickness.

Originality. The elaborated solution compositions make it possible to form stable oxide films of different composition, such as conversion coatings on steel at room temperatures. These oxide conversion coatings are resistant to corrosion in the majority of aggressive liquid and aerated environments. The applied environmental-friendly components shift the metal substrate into a passive state, providing additional corrosion resistance. The protective oxide coat is formed as a result of reduction of modifying additives on the surface of the metal to the form of oxygen-containing compounds.

Practical value. The proposed safe conversion solutions are an alternative to ecologically hazardous chromium-containing mixtures holding their own protective effect compared to known organic inhibitors, but do not require complicated synthesis. The developed modified solutions also exclude the energy component during obtaining conversion coatings that leads to simplification of hardware design and cheapening of technological process of their obtaining.

Keywords: *conversion coating, passive film, modifying solution, oxoanion, modification, ecological safety, reduction, oxide films*

Introduction. The creation of new technologies leads to the search for new modified corrosion-resistant materials and methods for their protection against the destructive effects of aggressive media. Apart from the economic efficiency, it also is necessary to consider environmental safety while manufacturing and managing these materials. Recently, in connection with the economic crisis, instead of the traditional galvanic, conver-

sion coatings with a number of protective specific properties are becoming increasingly widespread and used.

The term “conversion coating” (CC) refers to the coating or protective layer formed on the surface of the metal substrate or its oxidation products with specially selected components of the conversion solution at a certain temperature. The traditional solutions for the CC deposition contain toxic chromium compounds and are formed at elevated structure-forming temperatures. In addition, such waste solutions require special expensive

methods of cleaning or recycling. That is why today, the interest in solutions of the so-called “cold working of metals” without the content of toxic chromates especially increases and has become an urgent research topic.

Analysis of the recent research and publications. Nowadays, coatings become more popular, which have not only improved performance characteristics and high corrosion resistance, but also do not require complex technological equipment for their deposition. It is also necessary to consider the importance of wastewater treatment in such technologies, since this process requires additional staff to be involved as well as equipment [1].

According to the most recent applied studies on this topic, CCs are deposited on steel in hot concentrated electrolytes. These electrolytes often comprise highly-concentrated toxic or aggressive compounds.

Passive films, formed on the surface of steel at immersion in convertible solutions which contain oxoanions, without external current and heating, are more inexpensive and simple, from the point of deposition technology. There are known methods for obtaining such films on the mild steels, using chromate and nitrite compounds. But such solutions are very toxic and environmentally hazardous. In addition, they are reported to promote pitting corrosion of metal surfaces during exploitation.

At the moment, the work is under way to find and replace these substances with molybdates, tungstates and permanganates [2].

These solutions are capable of protecting electro-negative metals – aluminum [3], magnesium, zinc and their alloys, cadmium coatings, and stainless steel [4].

There are no chromate-free electrolytes capable of being used for obtaining passive films that may serve as CC for the mild steel. In addition, most studies show that the obtained molybdate and other conversion coatings are inferior to performance properties of films obtained from the hot solutions. Therefore, the work on improvement of such modified coatings continues and remains highly relevant [5].

Objectives of the article. The purpose of this work was to study the process of protective film formation on the mild steels in passivating acetate based solutions with addition of ecologically safe oxoanions.

Methodology. The electrochemical measurements and corrosion tests were carried out using mild steel samples with the following composition, %: Fe – 99.34; C – 0.05; Si – 0.1; S – 0.035; Cu – 0.3; Ni – 0.15; P – 0.025.

The samples were polished by abrasive papers of different grit sizes, degreased with sodium carbonate, rinsed with distilled water, dried at room temperature.

Conversion coatings were obtained by exposing the samples to zinc-acetate-based solutions for 20–60 minutes at 18 ± 2 °C. Due to being less toxic than chromates and nitrites, ammonium and sodium molybdates, potassium bromate were chosen as modifying oxidizing additives.

The free-flow potentials of steel samples were measured right after their dipping in the acetate solutions

containing the additives of oxoanions and without them. The displacement of corrosion potential to the positive side per $300 \div 700$ mV in comparison with the potential of steel in an active condition testified to formation of a passive film.

The rating of a stability and protective action of the formed passive film was carried out as follows: the passivated samples were moved to the solution which is not containing additives of oxoanions – low-mineralized water ($0.319 \text{ g/dm}^3 \text{ Na}_2\text{SO}_4 + 0.3 \text{ g/dm}^3 \text{ NaHCO}_3$); then the displacement in time of corrosion potential and an anodic polarizing curve were measured. Changes in potential were measured for $10 \div 60$ minutes.

Both these state were verified by comparison of the free corrosion potential to the values derived from analysis of polarization curves, obtained on potentiodynamically polarized steel surface (scan rate $2 \mu\text{V/s}$). The samples' working area was 1 cm^2 .

The polarization investigations were performed by using the potentiostat (PI-50-1.1) and a PR-8 programmer, oscillograph (C 8-13). In order to control pH of the solution ionometer (EV-74) was employed.

The study was held in a regular 170-ml electrochemical cell. The mild steel potential was measured relative to the silver-chloride reference electrode. The obtained electrode potentials were recalculated relative to the normal hydrogen electrode.

The stability and protective properties of obtained CC was evaluated by corrosion tests in a 10 % solution of sodium chloride in a sealed testing chamber. The test process included cyclic warming and cooling of the samples in a corrosion chamber of varying humidity.

Corrosion rates and conversion coatings' effectiveness are calculated by means of the following equations

$$CR = \frac{W_0 - W_1}{S \cdot \tau}; \quad (1)$$

$$CCE = \frac{CR_1 - CR_2}{CR_1} \cdot 100\%, \quad (2)$$

where CR s is the rate of corrosion damage of coating, $\text{g} \cdot \text{m}^{-2}\text{h}^{-1}$; S is the sample area (m^2); W_0 is the initial weight of the sample; W_1 is the sample weight (g) after the testing period; τ is the exposure period (h), and CR_1 and CR_2 are the corrosion rates without and with conversion coating, respectively.

Compounds inclusion in passivating films and the surface morphology was verified with an electron microscope (REM-103).

Presentation of the main research. Zinc acetate, as the main component of modified convertive solutions, was used. Its passivating properties were studied in [6].

Steel is in an active state in the solution of zinc acetate ($7.5 \text{ g/dm}^3 \text{ ZnAc}$) without the additives of passivating oxoanions. The displacement of steel free corrosion potential in time in the negative direction ($E = -0.53 \text{ V}$ after 20–30 minutes of exposure) confirmed this.

In order to prevent zinc precipitation, acetic acid was added to the solution. pH of the solution did not exceed

Table 1

Steel stationary potential in the passivating and reference solutions

Solution	Passivating solution, g/dm ³					Passivation potential, E, V		Potential in low-mineralized water, E, V	
	ZnAc	NaAc	Na ₂ MoO ₄	(NH ₄) ₆ Mo ₇ O ₂₄	KBrO ₃	10 sec	20 min	10 sec	20 min
1	7.5	0	0	0	0	-0.53	-0.53	-0.336	-0.627
2	0	5.8	0	0	0	-0.61	-0.61	-0.528	-0.63
3	7.5	0	0	0	4	0.049	0.226	0.03	-0.332
4	7.5	0	4	0	3	-0.397	-0.341	-0.399	-0.4
5	7.5	0	4	0	4	-0.329	-0.297	-0.301	-0.091
6	7.5	0	3	0	4	-0.335	-0.238	-0.250	-0.317
7	0	5.8	3	0	4	-0.450	-0.354	-0.455	-0.047
8	7.5	0	0	8	4	-0.302	0.166	0.078	-0.177
9	0	5.8	0	8	4	-0.445	-0.363	-0.350	-0.083

3.5. Zinc acetate promotes steel passivation better than sodium acetate (Table 1).

The passivating action of zinc ions can be explained by the insoluble compounds formation. While accumulating, they block the metal surface and, as a result, inhibit its further corrosive destruction.

However, the acetate solutions without additives of oxoanions promote the formation of passive films that have no aftereffect. Such films are not stable to the influence of aggressive environments.

The addition of oxoanions to the zinc acetate solution causes the potential displacement in time in the positive direction. This indicates the steel transition into a passive state (Fig. 1).

The samples passivated in this way were transferred into the reference solution (low-mineralized water), where their stationary potentials were measured. Afterwards, the potentials on passivated and

non-passivated surfaces were measured and compared.

In low-mineralized water, the stationary potential of a steel sample without a preliminary passivation is shifted to the negative side. In five minutes, after dipping, the potential became constant $E_{st} = -0.648$ V (Fig. 2). This indicates the steel active state, while samples with pre-applied conversion coatings have more positive potential values under the same conditions. This proves that protective films persist on the samples' surfaces.

Samples with the passive films obtained in zinc-acetate solutions with potassium bromate additive have the stationary potential slump when put in the reference solution. This behavior gives evidence of the metal surface activation. In this way, potassium bromate does not form the stable passivation films. Although bromate is an effective passivator, it does not have aftereffect [7].

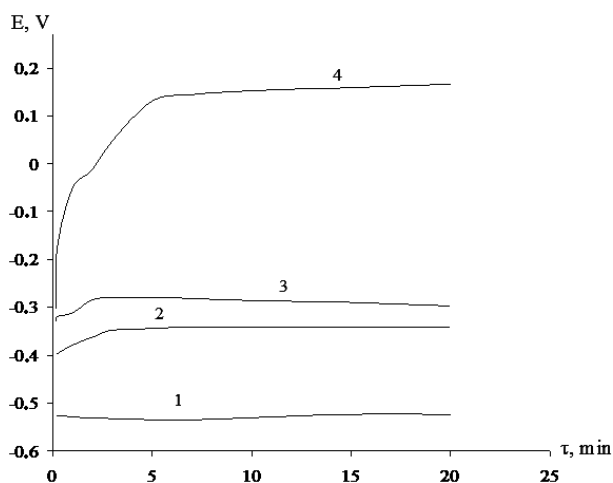


Fig. 1. Change of steel stationary potential in time during chemical passivation in zinc acetate solution with oxoanion additives:

concentration of oxoanion additives, g/dm³: 1 – 0; 2 – 4.0 Na₂MoO₄ + 3.0 KBrO₃; 3 – 4.0 Na₂MoO₄ + 4.0 KBrO₃; 4 – 8.0 (NH₄)₆Mo₇O₂₄ + 3.0 KBrO₃

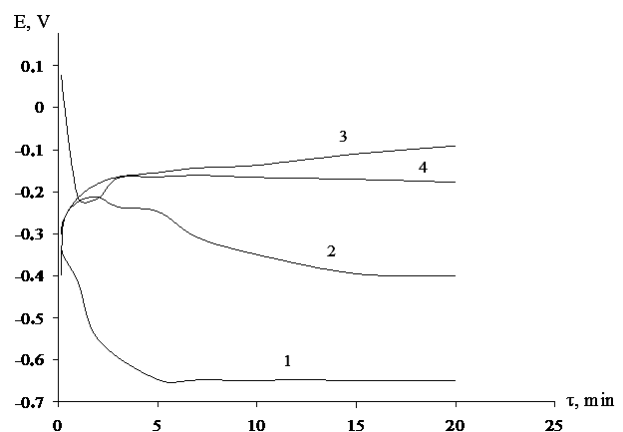


Fig. 2. Change of steel free corrosion potential in time in the low-mineralized water without passivation (1) and after passivation in the Zn(CH₃COO)₂ solution with oxoanion additives (2–4):

concentration of oxoanion additives, g/dm³: 1 – without preliminary passivation; 2 – 4.0 Na₂MoO₄ + 3.0 KBrO₃; 3 – 4.0 Na₂MoO₄ + 4.0 KBrO₃; 4 – 8.0 (NH₄)₆Mo₇O₂₄ + 3.0 KBrO₃

The instability of such passive films can be related with the appearance of bromate reduction products in the solution in the form of bromine ions, which have an activating effect [2].

The micro-X-ray analysis of the steel surface layer composition showed the bromate compounds inclusion in a passive film in very small quantities (Table 2). Obviously, the formed films contain unstable iron oxides, similar to those formed during the low-carbon steel anodic passivation [8].

Introducing molybdates to the passivating solution causes the steel potential shift in the positive direction, and makes it possible to obtain conversion coatings exhibiting a significant aftereffect in water without passivating additives.

It is shown that molybdates contribute to the passive film stabilization due to adsorption and its modification [9].

According to the previous scanning X-ray microscopy studies, molybdenum was discovered to be included in protective films as a ferric molybdate [9, 10].

It is also possible that obtained films are the mixture of products of incomplete molybdenum reduction to MoO_2 [9]. The presence of this or another oxide form in the passive film depends on the concentration of molybdate additives, pH of the solution and passivating treatment duration [11].

The exposure time has a great influence on the quality and properties of the formed oxide conversion coatings. Its increasing resulted in the growth of not only the content of oxoanions in the film (Table 2) but also of conversion layer thickness to the range of $0.1 \div 4.0 \mu\text{m}$ (Fig. 3). The film color became intense (from blue to

dark-purple) with an exposure time increase. It should also be noted that the surface aligned and became smoother during film growth (Fig. 4).

Protective properties of the formed CC are assessed based on the intensity of the steel surface corrosion damage after three months' exposure to 10 % NaCl solution fumes. Coatings, obtained from the zinc-acetate solutions with potassium bromate and ammonia molybdate additives, demonstrated the best results. Such a phenomenon can be explained by the content of polymerized molybdates in the formed modified oxide layers structures. Occurrence of dark purple color of the film which is characteristic of heptamolybdate ions confirms the polymerization process. This conversion coatings' efficiency against corrosion reaches 99.5–99.7 %.

In this way, stable coatings can be obtained if the conversion solution contains the following: the insoluble binder compound – zinc acetate; components that after being reduced are included to the conversion film – molybdates, and the passivating additive – potassium bromate. Such ecologically safe modified solutions allow obtaining stable coatings with fixed thickness and high corrosive protection efficiency.

Conclusions and recommendations for further research. The zinc acetate was proposed as the main component of the modified conversion mixtures. As modifying elements, potassium bromate, sodium and ammonium molybdate compounds have been used for conversion coating formation. The fact of the modifying component inclusion in the conversion films was verified by scanning X-ray microscopy.

The coatings were applied by dipping in the modifying solution at room temperature and keeping there for a

Table 2

Contents of modifying components in conversion films formed on the steel

№	Conversion solution composition, g/ dm^3	pH	Exposure time, min	Modifying component content in the film, mass %		
				Zn	Mo	Br
1	Na_2MoO_4 4.0 KBrO_3 3.0	8.7	20	–	8.44	0.08
2	$(\text{NH}_4)_2\text{Mo}_7\text{O}_{24}$ 8.0 KBrO_3 3.0	5.9	20	–	2.87	–
3	$\text{Zn}(\text{CH}_3\text{COO})_2$ 7.5 Na_2MoO_4 4.0 KBrO_3 3.0	3.5	20	0.25	4.44	0.08
4	$\text{Zn}(\text{CH}_3\text{COO})_2$ 7.5 Na_2MoO_4 4.0 KBrO_3 3.0	3.5	40	0.29	5.08	0.08
5	$\text{Zn}(\text{CH}_3\text{COO})_2$ 7.5 Na_2MoO_4 4.0 KBrO_3 3.0	3.5	60	0.31	5.26	0.06
6	$\text{Zn}(\text{CH}_3\text{COO})_2$ 7.5 $(\text{NH}_4)_2\text{Mo}_7\text{O}_{24}$ 8.0 KBrO_3 3.0	3.5	20	0.27	4.87	0.04
7	$\text{Zn}(\text{CH}_3\text{COO})_2$ 7.5 $(\text{NH}_4)_2\text{Mo}_7\text{O}_{24}$ 8.0 KBrO_3 3.0	3.5	40	0.28	5.94	0.06
8	$\text{Zn}(\text{CH}_3\text{COO})_2$ 7.5 $(\text{NH}_4)_2\text{Mo}_7\text{O}_{24}$ 8.0 KBrO_3 3.0	3.5	60	0.28	7.82	0.06

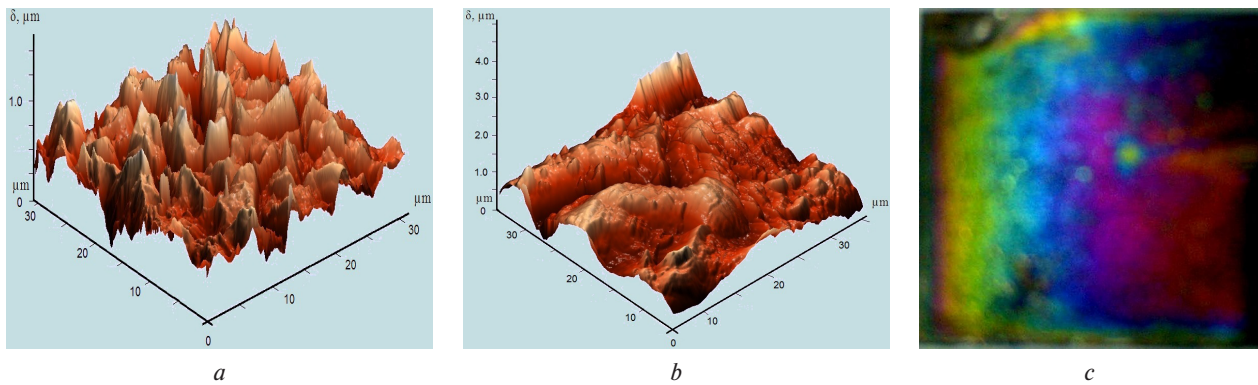


Fig. 3. Steel surface without (a) and with (b, c) acetate conversion coating

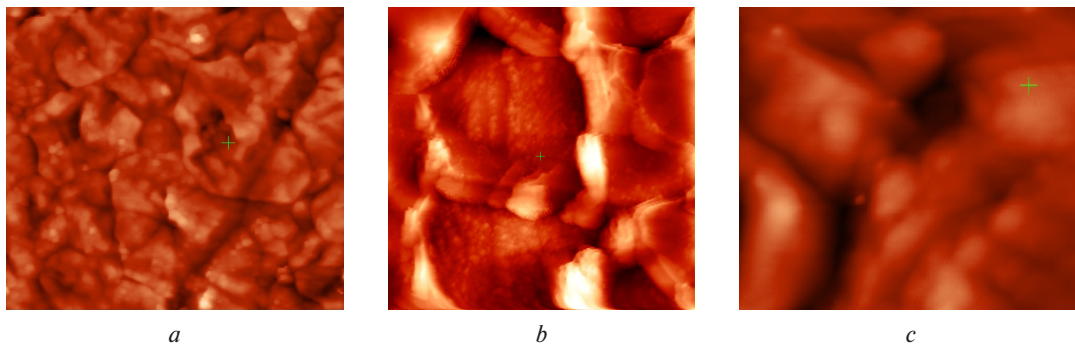


Fig. 4. Morphology change in the steel surface during the acetate conversion coating formation: exposure time, minutes: a – 10; b – 20; c – 60

certain time. It was noticed that the longer exposure time resulted in the film thickening (in the range of $0.1 \div 4.0 \mu\text{m}$) and additives contents in obtained layers.

Also, the advantages of the thin modified oxide passive films as the protective conversion coatings are in the fact that they do not change the properties of the steel surface, do not require the film to be removed for further operations and do not interfere with the next steel processing after storing under atmospheric conditions.

The designed solutions are able to form the stable films not only on steels of different composition, but also on zinc, aluminum, magnesium and their alloys. They allow obtaining smooth films on the metal surfaces, which are highly corrosion-resistant in the majority of the aggressive liquid and air environments. The corrosive protection efficiency of conversion coatings reaches 99.5–99.7 %.

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Екологічно безпечні модифіковані розчини для нанесення конверсійних покриттів на сталь

С. В. Фроленкова¹, В. І. Воробйова¹, Т. А. Оверченко¹, М. І. Скиба²

1 – Національний технічний університет України „Київський політехнічний інститут імені Ігоря Сікорського“, м. Київ, Україна, e-mail: svetlana.frolenkova@gmail.com

2 – Державний вищий навчальний заклад „Український державний хіміко-технологічний університет“, м. Дніпро, Україна, e-mail: Margaritaskiba@gmail.com

Мета. Аналіз впливу природи компонентів розчинів на закономірності процесів формування конверсійних покриттів на маловуглецевих сталях.

Методика. Нанесення покриттів здійснювали методом занурювання в модифікуючий розчин при температурі навколишнього середовища й витримки у ньому протягом певного часу. Для вивчення впливу компонентів конвертуючих розчинів на поведінку сталі застосовували потенціостатичні та потенціодинамічні поляризаційні виміри. Склад конверсійних плівок визначали за допомогою рентгенівського мікроаналізатора. Фотографії нанесених покриттів отримували на оптичному та електронному мікроскопах.

Результати. Розроблено ряд холодних розчинів для формування модифікованих оксидних конверсійних покриттів на маловуглецевій сталі для тимчасового захисту від корозії в рідких і повітряних середовищах. В якості основи для таких сумішей запропоновані розчини оцтовокислого цинку. Як модифікуючі добавки використовували оксоаніони, включення яких до складу оксидних конверсійних плівок підтверджено методом рентгенівської скануючої мікроскопії. Встановлено, що збільшення часу витримки зразка в пасивуючому розчині призводить до підвищення вмісту модифікуючих компонентів у плівці та росту її товщини.

Наукова новизна. Розроблені склади розчинів дозволяють формувати на маловуглецевій сталі за кімнатних температур стійкі оксидні плівки, типу конверсійних покриттів, що проявляють підвищену корозійну стійкість у більшості агресивних рідких і повітряних середовищ. Підібрані екологічно безпечні компоненти переводять метал-основу до пасивного стану, забезпечуючи додаткову його стійкість до корозійного впливу. Наразі, сам захис-

ний оксидний шар формується в результаті процесів відновлення модифікуючих добавок на поверхні металу до кисневмісних сполук.

Практична значимість. Запропоновані безпечні конвертуючі розчини є альтернативою екологічно небезпечним хромвмісним сумішам, не поступаючи захисною дією органічним інгібіторам, що ще й потребують спеціального технологічного синтезу та випускаються промисловістю. Також розроблені модифікуючі розчини дозволяють виключити енергетичну складову процесу нанесення конверсійних шарів, що призводить до спрощення апаратурного оформлення та здешевлення технологічного процесу отримання таких покриттів.

Ключові слова: конверсійне покриття, пасивна плівка, модифікуючий розчин, оксоаніон, модифікація, екологічна безпека, відновлення, оксидні плівки

Экологически безопасные модифицированные растворы для нанесения конверсионных покрытий на сталь

С. В. Фроленкова¹, В. И. Воробьева¹, Т. А. Оверченко¹, М. И. Скиба²

1 – Национальный технический университет Украины „Киевский политехнический институт имени Игоря Сикорского“, г. Киев, Украина, e-mail: svetlana.frolenkova@gmail.com

2 – Государственное высшее учебное заведение „Украинский государственный химико-технологический университет“, г. Днепр, Украина, e-mail: Margaritaskiba@gmail.com

Цель. Анализ влияния природы компонентов растворов на закономерности процессов формирования конверсионных покрытий на малоуглеродистых сталях.

Методика. Нанесение покрытий осуществляли методом погружения в модифицирующий раствор при температуре окружающей среды и выдерживании в нём в течение определённого времени. Для изучения влияния компонентов конвертирующих растворов на поведение стали применяли потенциостатические и потенциодинамические поляризационные измерения. Состав конверсионных плёнок определяли при помощи рентгеновского микроанализа. Фотографии нанесённых покрытий получали на оптическом и электронном микроскопах.

Результаты. Разработан ряд холодных растворов для формирования модифицированных оксидных конверсионных покрытий на малоуглеродистой стали для временной защиты от коррозии в жидких и воздушных средах. В качестве основы для таких смесей предложены растворы уксуснокислого цинка. Как модифицирующие добавки использованы оксоанионы, включение которых в состав оксидных конверсионных плёнок подтверждено методом рентгеновской сканирующей микроскопии. Установлено, что увеличение времени выдержки приводит к повышению содержа-

ния модифицирующих компонентов в плёнке и росту её толщины.

Научная новизна. Разработанные составы растворов позволяют формировать на малоуглеродистой стали при комнатных температурах устойчивые оксидные плёнки, типа конверсионных покрытий, проявляющие повышенную коррозионную стойкость в большинстве агрессивных жидких и воздушных сред. Подобранные экологически безопасные компоненты переводят металл-основание в пассивное состояние, обеспечивая дополнительную его устойчивость к коррозионному воздействию. Сам же защитный оксидный слой формируется в результате восстановления модифицирующих добавок на поверхности металла до кислородсодержащих соединений.

Практическая значимость. Предложенные безопасные конвертирующие составы являются альтер-

нативой экологически опасным хромсодержащим смесям, не уступая в защитном действии известным органическим ингибиторам, и, в отличие от последних, не требуют специального технологического синтеза, а выпускаются промышленностью. Также разработанные модифицированные растворы позволяют исключить энергетическую составляющую процесса нанесения конверсионных слоёв, что приводит к упрощению аппаратного оформления и удешевлению технологического процесса получения таких покрытий.

Ключевые слова: конверсионное покрытие, пассивная плёнка, модифицирующий раствор, оксоанион, модификация, экологическая безопасность, восстановление, оксидная плёнка

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