Protection of zinc, galvanized steel and "galvanic zinc–steel" contact couples by chamber corrosion inhibitors. Authors' review

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Abstract

The review summarizes the authors' studies on the chamber protection of zinc, galvanized steel, and "galvanic zinc–steel" contact couples. It was found that effective chamber protection of zinc is provided by α-branched carboxylic acids such as ethylhexanoic acid (EHA) and neodecanoic acid (NDA). Zinc and galvanized steel have a similar but not identical corrosion-electrochemical behavior. Nevertheless, the optimum temperature and duration of chamber treatment of these materials with EHA and NDA are the same. Nano-sized polymolecular adsorption films are formed on the metal surface under optimal conditions of chamber treatment of zinc and galvanized steel in EHA and NDA vapors. They passivate the metals and stabilize their passive state in chloride-containing media. These films inhibit the thermal oxidation of zinc and are capable of structuring on exposure of electrodes in the air during one day after chamber treatment. This structuring is accompanied by a growth in zinc protection efficiency. It has been found that the basic salt $C_4H_9–CH(C_2H_5)–COO–Zn–OH$ is formed on the metal in the course of chamber treatment. Dehydrogenation of the salt takes place upon exposure in the air with formation of the compound $C_4H_9–CH(C_2H_5)–COO–Zn–O–Zn–OOC–CH(C_2H_5)–C_4H_9$, which provides zinc protection. The protection of zinc and galvanized steel from corrosion by EHA and NDA occurs by a blocking mechanism. Protective films formed during chamber treatment of zinc with EHA and NDA have the self-healing ability in the case of mechanical damage to their integrity. Effective simultaneous protection of zinc and carbon steel is provided by mixtures of EHA with urotropine or octadecylamine. Treatment of samples with EHA and NDA vapors can be used for temporary protection of zinc and galvanized steel products. It is expedient to use mixtures of EHA with octadecylamine or urotropine for temporary protection of "galvanic zinc–steel" contact couples.

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Introduction

Corrosion losses amount to trillions of dollars per year [1]; 40–60% of these losses are due to the destruction of metals under weathering conditions [2, 3].

Carbon steels are the most common metallic structural materials, therefore, their protection from atmospheric corrosion is an important scientific and technical task. Zinc coatings, including galvanic ones [4–7], are often used to solve this problem. The coatings isolate steel from atmospheric corrosive agents and provide its sacrificial protection in the surface moisture. The high efficiency of these coatings results in the fact that the corrosion of the substrate steel ceases to limit the service life of the products. The service life is determined by the corrosion resistance of the zinc coating. Cr(VI) compounds were widely used in the previous century to increase corrosion resistance [8], but now their use is limited due to toxicity. The efforts of specialists around the world are aimed at developing methods for the protection of galvanized steel that are comparable with chromate treatment in terms of price, technological performance, and efficiency. Cr(III) salts, phosphatization, water-soluble polymerizing formulations, etc. are used for zinc passivation [8, 9]. However, the problem of temporary protection of galvanized steel has not been fully solved and is still relevant today.

Certain achievements in this field are associated with the use of volatile inhibitors [10–17]. From the practical point of view, the approach based on the application of another type of vapor-phase inhibitors – chamber corrosion inhibitors (CIN) [18] – shows promise. The main aspects of this chamber protection (CP) method are as follows:

- by increasing the temperature in a closed space (chamber) it is possible to achieve an increase in the vapor pressure of inhibitors, which are low-volatile under normal conditions, to a level that ensures rapid formation of protective adsorption layers on the metal from the gas phase;
- when the temperature drops and the system is depressurized, the adsorption layers slowly evaporate from the surface due to the low vapor pressure of corrosion inhibitors, providing a long-lasting protective after-effect;
- with proper selection of the inhibitor and chamber treatment (CT) conditions it is possible to achieve efficient metal protection due to the anticorrosive after-effect.

The method of chamber protection of metals was proposed at the Institute of physical chemistry and electrochemistry of the Russian Academy of Sciences (IPCE RAS) seven years ago and it has been intensely developed there in the recent years [19, 20]. No work in this field has been carried out so far in other scientific institutes. The results of the studies indicate a significant potential of the CP method [20–74]. From the author’s point of view, the data on the use of this method for galvanized steel protection required aggregation and generalization. All this prompted the publication of this article and its composition as an author’s review.

Galvanized steel is a complex system for studying. The development of galvanized steel corrosion inhibitors was greatly facilitated by the use of zinc as an object of study at the first stage of this work. However, the corrosion-electrochemical behavior of zinc and galvanized steel is not identical. Therefore, the main conclusions of the studies obtained on bulk zinc were cross-checked on galvanic zinc.

In practice it is often necessary to protect products containing “galvanic zinc–steel” contact couples from atmospheric corrosion. Sometimes the presence of such couples is defined by the design of the items. They can also appear due to failures in galvanizing of items with a complex configuration.

All the considerations mentioned above determine the structure of this review. The prospects, specific features and mechanisms of corrosion protection of zinc, galvanized steel, and “galvanic zinc–steel” contact couples described in [37, 39, 47, 54–56, 62–65] are considered in the review step-by-step.

Chamber protection of zinc

At the first stage of this work, screening of the protective after-effects of individual compounds and mixed zinc corrosion inhibitors were under conditions of periodic condensation of moisture was performed (Table 1). The first signs of zinc corrosion appeared in the samples in the initial state, one hour after the beginning of the experiment. Thermal treatment without corrosion inhibitors did not affect the corrosion resistance of zinc. The incubation time of corrosion (protection time, τ_{prot}) still did not exceed 1 h.

Table 1. Protective after-effect of adsorption films formed during chamber treatment of zinc and steel with different corrosion inhibitors. Coefficients of mutual effect of components of mixed corrosion inhibitors (α). Conditions: periodic condensation of moisture, chamber treatment temperature (t_{CT}) 120°C, chamber treatment duration (τ_{CT}) 1 h, time of exposure in the air after chamber treatment (τ_{exp}) 24 h.

Treatment conditions, corrosion inhibitors	**τ_{prot}, h/α**	
	Zinc	**Steel**
Initial state	1.0/–	1.0/–
Thermal treatment without corrosion inhibitors	1.0/–	1.0/–
Octadecylamine	4.0/–	129.0/–
Diphenylguanidine	2.0/–	10.0/–
Polyethylene polyamine	12.0/–	7.0/–
Urotropine	5.0/–	2.0/–
Captax	4.0/–	1.0/–
Altax	2.0/–	1.0/–
Benzotriazole	8.0/–	3.0/–
Lauric acid	24.0/–	24.0/–
Tridecanoic acid	48.0/–	4.0/–
Stearic acid	24.0/–	1.0/–
Oleic acid	12.0/–	4.0/–
Linolenic acid	12.0/–	3.0/–

Treatment conditions, corrosion inhibitors	**τ_{prot}, h/α**	
	Zinc	**Steel**
2-Ethylhexanoic acid (EHA)	1680.0/–	12.0/–
Neodecanoic acid (NDA)	1632.0/–	2.0/–
Stearic acid with urotropine	72.0/0.2	504.0/252
Linolenic acid with urotropine	24.0/0.4	504.0/84
Tridecanoic acid with urotropine	144.0/0.6	48.0/6
NDA with urotropine	504.0/0.06	24.0/6
EHA with urotropine	840.0/0.1	504.0/21
EHA with polyethylene polyamine	264.0/0.01	24.0/0.3
Tridecanoic acid with polyethylene polyamine	72.0/0.1	1.0/0.04
NDA with octadecylamine	504.0/0.08	24.0/0.09
Tridecanoic acid with octadecylamine	3.0/0.02	3.0/0.006
EHA with octadecylamine	336.0/0.05	216.0/0.01
Octadecylamine with benzotriazole	6.0/0.2	168.0/0.4
Octadecylamine with Captax	2.0/0.1	2.0/0.02
Tridecanoic acid with benzotriazole	48.0/0.1	48.0/4

All the substances studied inhibited corrosion initiation to some extent. The greatest protective effect was observed for carboxylic acids with α-branched hydrocarbon chain, namely ethylhexanoic acid (EHA) and neodecanoic acid (NDA). They provided full zinc protection for 1680 and 1632 h, respectively. Other corrosion inhibitors, including mixed ones, were inferior to these compounds in protective after-effect, although formulations based on EHA and NDA significantly (250-fold or more) inhibited corrosion initiation.

The mixed corrosion inhibitors under study were characterized by values $\alpha<1$ reflecting an antagonism of protective action that can be related to the acid-base interactions of the components. These interactions decrease the vapor pressure of inhibitors and their adsorption on the metal.

It has been shown earlier that each "metal–corrosion inhibitor" system is characterized by optimal values of the chamber treatment temperature (t_{CT}) and chamber treatment duration (τ_{CT}). It was found in tests under salt fog conditions that the "t_{CT}–τ_{prot}" plot for EHA has a maximum at 100°C. The upward branch of the plot is associated with an increase in the vapor pressure of the inhibitor, which favors adsorption, while the downward branch is associated with a decrease in vapor adsorption upon heating of the adsorbent.

The protective after-effect first increased and then stabilized with an increase in τ_{CT}. A further increase in τ_{CT} did not increase the protective after-effect and, therefore, it was useless.

In the further study of the protective properties of EHA, adsorption films on zinc were formed at optimal t_{CT} and τ_{CT}. They were not inferior to the films formed during zinc chromate treatment in terms of zinc protection efficiency in salt spray (72 hours in both cases).

Polarization methods showed that chamber treatment of zinc with EHA inhibited zinc anodic dissolution, suppressing the active dissolution and stabilizing the passive state in chloride-containing electrolytes (Figure 1). The passivation current densities (i_{pas}) decreased and the pitting potentials (E_{pit}) increased during the first day of exposure of zinc electrodes in the air after the chamber treatment (τ_{exp}).

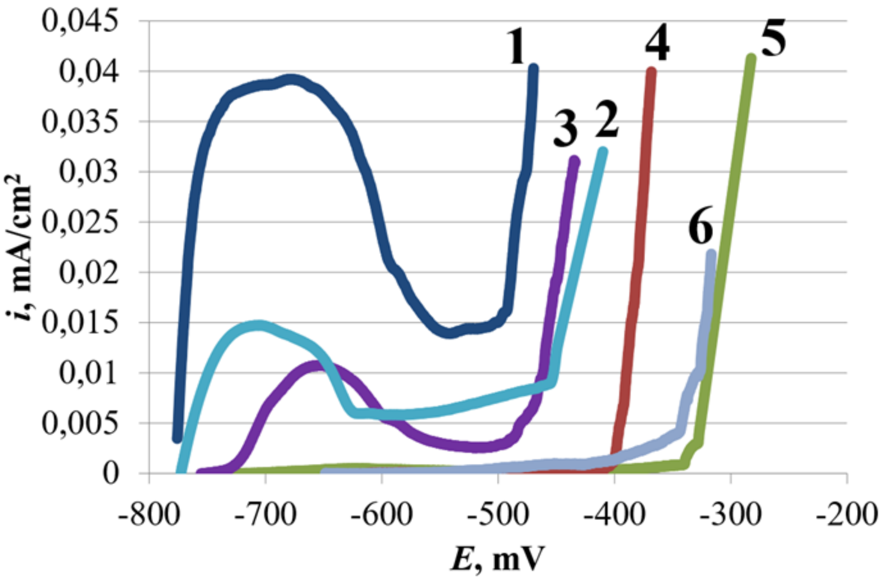

Figure 1. Anodic polarization curves of zinc. Borate buffer, 1 mM, pH 7.36; t_{CT} = 100°C, τ_{CT} = 1 h. 1 – initial state; 2 – after thermal treatment without inhibitors; τ_{exp} = 24 h. Chamber treatment in EHA vapors at different τexp: 3 – 1 h; 4 – 5 h; 5 – 24 h; 6 – 72 h.

Electrochemical impedance spectroscopy (EIS) provides additional information on the mechanism of zinc protection by EHA. Nyquist plots of zinc electrodes at different variants of treatment are shown in Figure 2.

All the hodographs given in this paper had the shape of deformed half-circles in which two capacitive loops could be distinguished. They can be described by the equivalent circuit shown in Figure 3.

The radius of arcs increased many times after chamber treatment of zinc with EHA. The protective properties increased symbatically with τ_{exp} during one day.

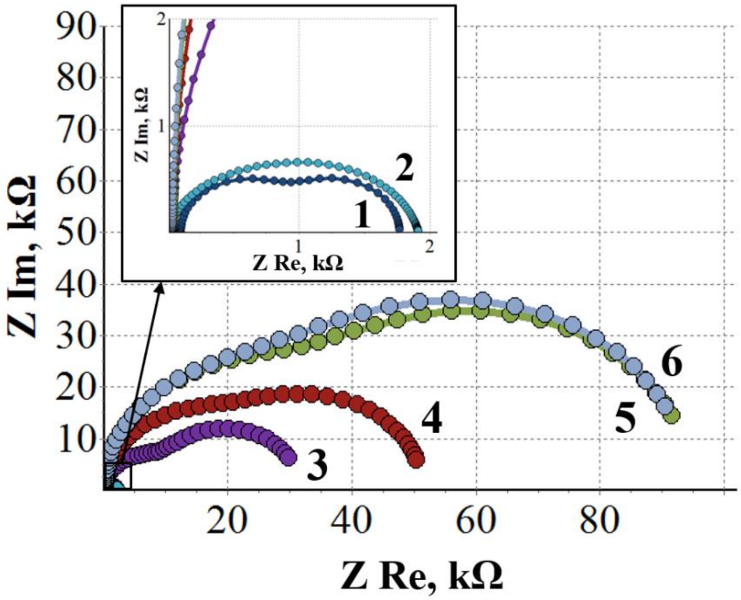

Figure 2. Nyquist plots of zinc. Borate buffer, 1 mM, pH 7.36; $t_{CT}=100$°C, $\tau_{CT}=1$ h. The numbering of curves is identical to Figure 1.

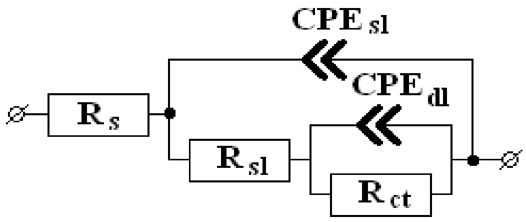

Figure 3. Equivalent circuit for the hodographs. R_s is the resistance of the electrolyte between the working electrode and the capillary of the reference electrode, R_{sl} is the total resistance of surface layers, R_{ct} is the charge transfer resistance for the reaction that determines the corrosion kinetics, CPE_{sl} and CPE_{dl} are the constant phase elements characterizing the capacitances of the metal-electrolyte interface and the electric double layer, respectively.

The parameters of the equivalent circuit were determined and the characteristic values of the protective effect of the corrosion inhibitor were calculated. Their values are given in Table 2: γ_{bl} and γ_{act} are the corrosion inhibition coefficients by the blocking and activation mechanism, Θ is the degree of surface coverage with corrosion-blocking layers, and Z is the degree of metal protection.

Thermal treatment of the electrode in the absence of corrosion inhibitors was not accompanied by noticeable protection; in contrast, chamber treatment inhibited corrosion efficiently. Changing of the time of exposure in the air after chamber treatment (τ_{exp}) from 1 to 24 h led to an increase in γ_{bl} almost 50-fold. At the same time, the degree of surface coverage Θ increased up to 98%. The coinciding values of Θ and Z and the γ_{act} value close to unity indicate a blocking mechanism of EHA action. The protective properties of the adsorption films stabilized in one day after the chamber treatment.

Table 2. Characteristic values of protective effect of EHA at different variants of zinc treatment. Numbering of experiments is identical to Figure 1.

No.	Treatment conditions, inhibitor	γ_{bl}	γ_{act}	Θ, %	*Z*, %
2	Thermal treatment without corrosion inhibitors, τ_{exp}=24 h	1.10	1.00	9	9
3	EHA, τ_{exp}=1 h	13.4	2.01	92	96
4	EHA, τ_{exp}=5 h	30.7	1.00	97	97
5	EHA, τ_{exp}=24 h	49.7	1.36	98	98
6	EHA, τ_{exp}=72 h	49.5	1.14	98	98

According to ellipsometry data (Table 3), surface blocking and zinc protection occurred due to formation of surface films up to 105 nm thick. Chamber treatment inhibited the thermal oxidation of zinc and caused a decrease in the thicknesses of oxide layers. The growth in τ_{exp} was accompanied by thinning of EHA films. Since the length of the EHA molecule does not exceed 1 nm, it can be stated that its adsorption layers were polymolecular.

Table 3. Surface film thicknesses upon zinc chamber treatment in different variants, t_{CT}=100°C, τ_{CT}=1 h. The numbering of experiments is identical to Figure 1.

No.	Treatment conditions, inhibitor	Thickness, nm	
		Oxide film	Adsorption film
2	Thermal treatment without corrosion inhibitors	5.5	–
3	EHA, τ_{exp}=1 h	1.0	105
4	EHA, τ_{exp}=5 h	1.0	95
5	EHA, τ_{exp}=24 h	1.0	85

An important fact is a peculiar self-organization of EHA adsorption layers upon exposure in the air after chamber treatment. The mechanism can be studied by X-Ray Photoelectron Spectroscopy (XPS). Figure 4 shows the C1s, O1s XPS spectra and the $ZnL_3M_{45}M_{45}$ Auger spectra for zinc after zinc surface treatment by different variants.

Zinc surface in the initial state contain impurities. This is evidenced by the C1s and O1s electron spectra. The spectra contained peaks related to saturated hydrocarbon chains and groups of atoms containing both carbon and oxygen. In addition, peaks of adsorbed water and hydroxide and zinc oxides were present in the spectra. The $ZnL_3M_{45}M_{45}$ Auger spectra of the sample in the initial state are approximated by peaks corresponding to zinc, zinc oxide, and zinc hydroxide.

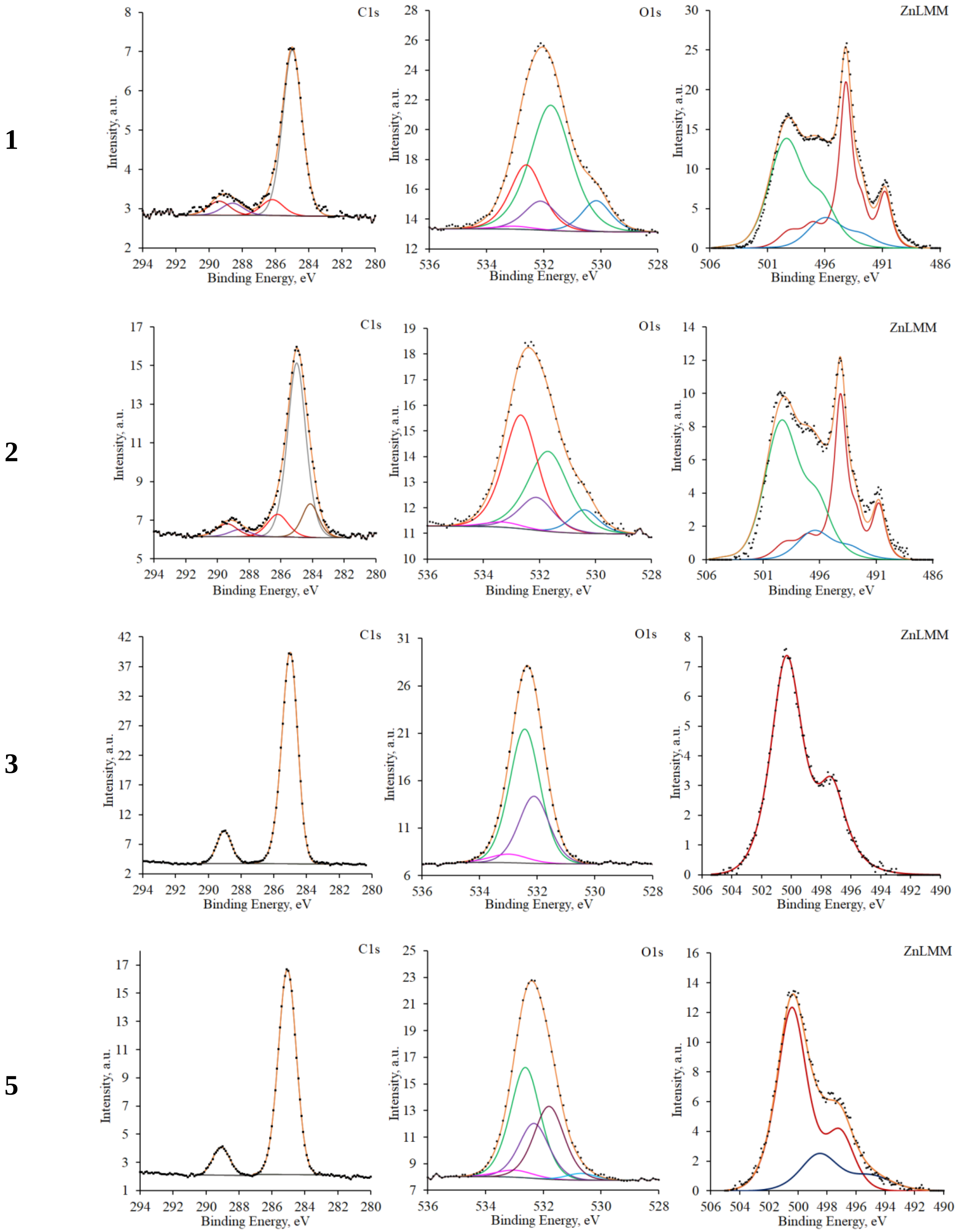

Figure 4. C1s, O1s XPS spectra and ZnL$_3$M$_{45}$M$_{45}$ Auger spectra for zinc at different variants of zinc surface treatment. The numbering of experiments is identical to Figure 1.

Peaks related to contaminants were also present on the zinc surface after the thermal treatment. In addition to the previously determined carbon states, a peak of C=C moieties appeared in the spectrum. The same states as those on zinc in the initial state were observed in the XPS spectrum of oxygen and Auger spectrum of zinc, but the atomic concentrations of the spectrum fractions changed.

No substrate peaks were observed in the spectra after the chamber treatment. The film thickness significantly exceeded the method's penetration depth. Two well-separated peaks of carbon atoms of the hydrocarbon chain and the carboxyl group were present in the spectra for samples with $\tau_{\mathrm{exp}}=1$ h. The ratio of integral intensities of the peaks was 7:1, which is consistent with the stoichiometry of EHA. The O1s spectrum of oxygen contained three peaks. One peak corresponded to adsorbed water and the other two, to hydroxyl and carboxyl group atoms. The intensity ratio of these peaks was 1:2. The $\mathrm{ZnL_3M_{45}M_{45}}$ Auger spectrum had two peaks that were typical of $\mathrm{Zn^{2+}}$ compounds and corresponded to one state of zinc.

Taking into account the sensitivity coefficients of the spectra described above, it is possible to conclude that a compound is present on the zinc surface after chamber treatment at $\tau_{\mathrm{exp}}=1$ h, which contains 7 carbon atoms of the hydrocarbon chain, 1 carbon atom and 2 oxygen atoms of the carboxyl group, and 1 oxygen atom of the hydroxyl group per 1 zinc atom. It is very likely to be the basic salt of zinc and EHA with the formula $\mathrm{C_4H_9{-}CH(C_2H_5){-}COO{-}Zn{-}OH}$.

The shape of the spectrum, the positions of maxima, and the ratio of intensities of carbon peaks did not change for the samples with $\tau_{\mathrm{exp}}=24$ h. This means that the hydrocarbon radical $\mathrm{C_4H_9{-}CH(C_2H_5){-}}$ did not undergo any changes when the samples were exposed in the air. In contrast, the spectra of oxygen and zinc did change on such exposure. Two peaks appeared in the spectrum of oxygen, which corresponded to the bridging oxygen in the $\mathrm{{-}Zn{-}O{-}Zn{-}}$ moiety and to the oxygen atoms of carboxyl groups, the electron density of which is different from the groups described above. In addition to the state determined at $\tau_{\mathrm{exp}}=1$ h in the $\mathrm{ZnL_3M_{45}M_{45}}$ Auger spectrum for the sample with $\tau_{\mathrm{exp}}=24$ h, one can identify the peaks of zinc atoms of the compound $\mathrm{C_4H_9{-}CH(C_2H_5){-}COO{-}Zn{-}O{-}Zn{-}OOC{-}CH(C_2H_5){-}C_4H_9}$, which is formed on dehydration of the basic salt of zinc and EHA. This reaction causes the protection efficiency growth.

A desirable property of protection coatings on metals is the self-healing of damage to their integrity. The self-healing of EHA adsorption films was evaluated by the drop method after a scratch was applied to the zinc surface. Colored corrosion products under a drop of lead acetate indicator solution appeared in less than a second on zinc in the initial state and after thermal treatment. Chamber treatment of zinc in EHA vapor inhibited corrosion. For the samples exposed in the air after chamber treatment for $\tau_{\mathrm{exp}}=24$ and 48 h, the drop color changed in 35 seconds. Application of the scratch violated the protective properties of the adsorption coating. The coloring of the drop on the scratch changed immediately, in fact instantly, after the scratch application. A day later, the protective properties of EHA layers inside the scratch were partially restored. The protection time τ_{prot} increased to 20 sec, indicating the self-healing of coatings.

The high protective properties of EHA with respect to zinc were confirmed by field tests at the Moscow corrosion station. Metal tarnishing was observed after one-week testing of zinc in the initial state and after thermal treatment without inhibitors. Chamber treatment with EHA provided metal protection for more than 42 months. These results give reasons for using chamber treatment with EHA, at least for the temporary protection of zinc items.

According to the data of Table 1, NDA is the second most effective compound for zinc chamber protection. The optimal conditions for the chamber treatment in the "NDA–zinc" system were determined at the beginning of the study. Experiments in salt fog showed that the optimum chamber treatment temperature t_{CT} for this system is 100°C. Lower or higher t_{CT} led to the formation of adsorption films with poorer protective properties. The optimum chamber treatment duration τ_{CT} was 1 h at t_{CT}=100°C. A further increase in τ_{CT} was not accompanied by an improvement in protective properties. Note that the optimal conditions for the chamber treatment of zinc by EHA and NDA coincide. However, the efficiency of NDA in salt spray (τ_{prot}=120 h) was superior to that of EHA and chromate treatment of zinc (72 h).

The mechanism of protective action of NDA was studied by potentiodynamic anodic polarization (Figure 4). The results indicate that chamber treatment of zinc with NDA passivates the metal and ennobles the pitting potential E_{pit}. E_{pit} grew symbatically with the time of exposure τ_{exp} during the first day of exposure of the electrodes in the air after chamber treatment. Comparing NDA and EHA, one may note that their effect on E_{pit} is commensurable, but NDC is inferior to EHA in terms of the anti-pitting basis.

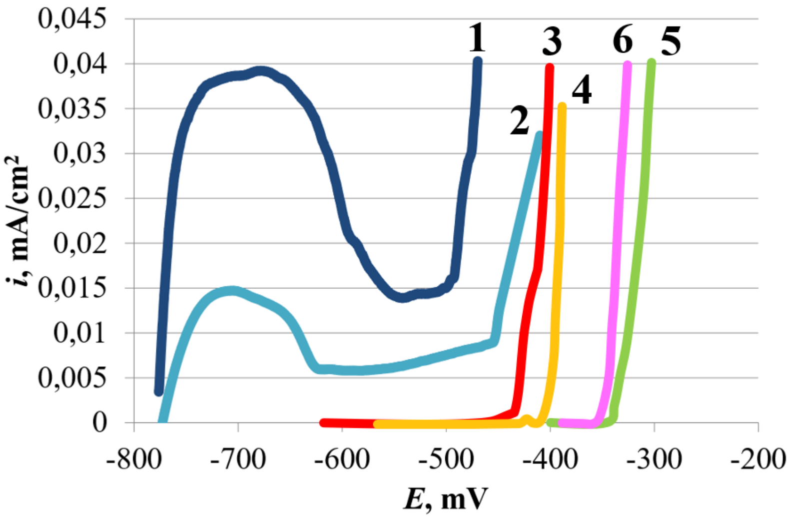

Figure 5. Anodic polarization curves of zinc. Borate buffer, 1 mM, pH 7.36, t_{CT}=100°C; τ_{CT}=1 h. 1 – zinc in the initial state; 2 – zinc after thermal treatment without inhibitors, τ_{exp}=24 h. Chamber treatment in NDA vapors at different τ_{exp}: 3 – 1 h; 4 – 5 h; 5 – 24 h; 6 – 72 h.

At the next step, the "NDA–zinc" system was studied by the impedance method. Nyquist plots of zinc after its treatment by different variants are shown in Figure 6. The radius of the hodographs increased when zinc was treated in NDA vapors. The resistive properties of the metal surface increased symbatically with τ_{exp} during one day.

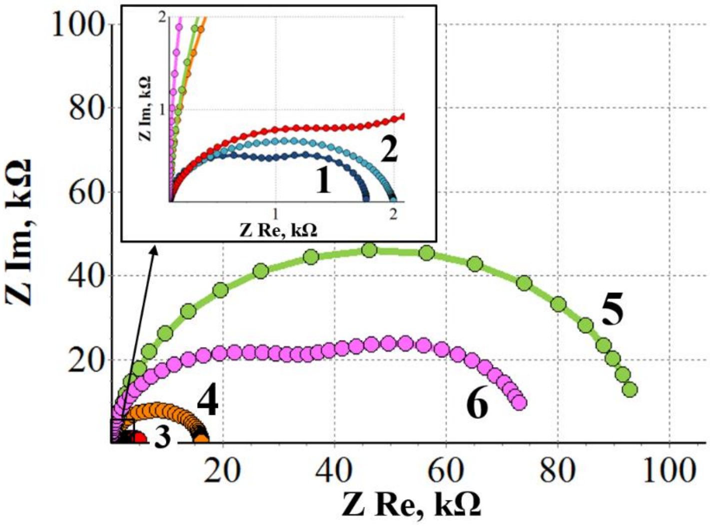

Figure 6. Nyquist plots of zinc. Borate buffer, 1 mM, pH 7.36, t_{CT} = 100°C, τ_{CT} = 1 h. The numbering of experiments is identical to Figure 5.

The calculated values of equivalent circuit elements and protective action characteristics of NDA are shown in Table 4.

Table 4. Characteristic values for NDA protective effect at different variants of zinc treatment. The numbering of experiments is identical to Figure 5.

No.	**Treatment conditions, inhibitor**	γ_{bl}	γ_{act}	**Θ, %**	***Z*, %**
2	Thermal treatment without corrosion inhibitors, τ_{exp} = 24 h	1.1	1	9	9
3	NDA, τ_{exp} = 1 h	1.8	2.94	44	81
4	NDA, τ_{exp} = 5 h	7.7	1.61	87	92
5	NDA, τ_{exp} = 24 h	20.1	5.51	95	99
6	NDA, τ_{exp} = 72 h	42.8	1.13	98	98

Chamber treatment of the electrode with NDA resulted in corrosion inhibition. The metal protection degree *Z* varied from 9 (zinc after chamber treatment) to 99%. *Z* values increased symbatically with τ_{exp} during the first day of exposure in the air after chamber treatment, but longer τ_{exp} were not accompanied by an increase in *Z*. Comparison of the data in Tables 2 and 4 indicates that EHA and NDA have similar efficiency. Comparison of the corrosion inhibition coefficients by the blocking and activation mechanisms, γ_{bl} and γ_{act}, indicates that the corrosion inhibitor acts by the blocking mechanism. At optimum conditions of metal treatment and $\tau_{exp} \geq 24$ h, the degrees of surface coverage with corrosion-blocking layers Θ were close to 1.

The thicknesses of NDA adsorption films on zinc could not be measured by ellipsometric methods because of strong scattering of the laser beam. This usually happens at thicknesses of surface layers larger than 100 nm. Taking into account the linear size of NDA (no more than 1 nm), this fact indirectly indicates the polymolecular nature of the surface layers.

Like in the case of EHA, the films formed on zinc in NDA vapor are capable of self-healing of mechanical damage to their integrity. This is evidenced by the results of drop tests. Chamber treatment of zinc in NDA vapors increased the time of color change of the indicator solution to 3 s. Mechanical damage of the coating integrity impaired its protective properties. The color of the drop on a scratch changed immediately, in fact instantly, after its application, similarly to the zinc surface that did not undergo chamber treatment. A day later, the protective properties of the adsorption layers inside the scratch restored completely.

The conditions of zinc corrosion under a drop of the indicator solution differ strikingly from the atmospheric corrosion conditions. The shorter time of color change of the indicator drop on zinc treated with NDA in comparison with that of EHA does not indicate that NDA is a less efficient corrosion inhibitor.

Field tests were conducted at the last stage of the study of NDA's protective properties. This inhibitor provided metal protection for more than 24 months. The experiment was continuing at the time this review was written. These data allow NDA to be recommended for the temporary protection of zinc items.

Chamber protection of galvanized steel

The specific features of protection of galvanized steel by EHA and NDA vapors were then studied. Optimal conditions for the chamber treatment by these inhibitors were determined in salt fog where galvanized steel is subject to "white" corrosion. Corrosion sites appeared on the specimens during the first hour of tests. Thermal treatment had no effect on the corrosion resistance of galvanized steel.

Chamber treatment with EHA slowed down the initiation of galvanized steel corrosion markedly. The optimum treatment conditions were 100°C and 1 h. Galvanized steel treated with EHA had better corrosion resistance than zinc subjected to the same treatment (96 and 72 h, respectively).

The anodic polarization curves of galvanized steel and zinc in the initial state and after thermal treatment are similar (Figure 7). The regions of active dissolution, passivity, and pitting can be seen on the curves. However, the characteristics of the curves differed. The passivation current densities i_{pas} for galvanized steel were lower than those for zinc. Galvanized steel also differed from zinc by greater resistance to pitting.

Chamber treatment in EHA vapors passivated galvanized steel and stabilized its passive state. Exposure of galvanized steel electrodes in air after treatment with EHA was accompanied by structurization of the adsorption films. The protective effect of EHA increased during one day.

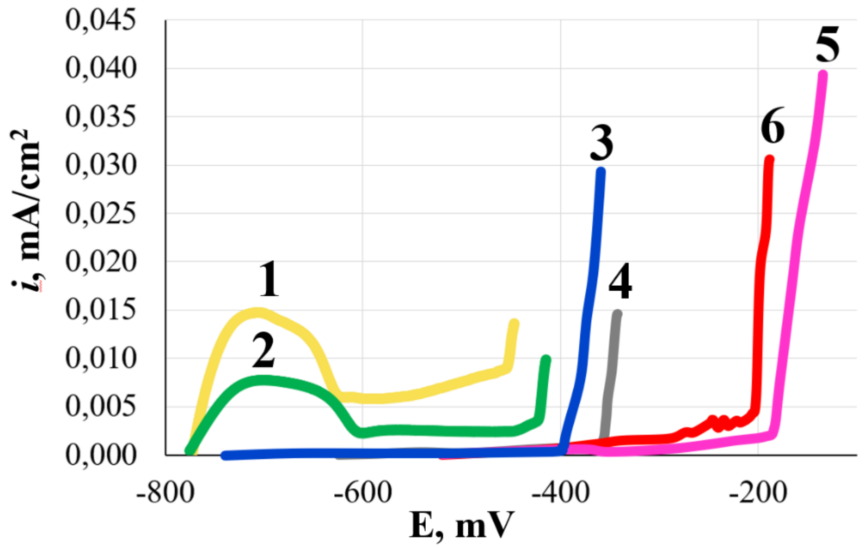

Figure 7. Anodic polarization curves of galvanized steel. Borate buffer, 1 mM, pH 7.36; t_{CT}=100°C; τ_{CT}=1 h. 1 – samples in the initial state; 2 – samples after thermal treatment without corrosion inhibitors, τ_{exp}=24 h. Chamber treatment in EHA vapors at different τ_{exp}: 3 – 1 h; 4 – 5 h; 5 – 24 h; 6 – 48 h.

The mechanism of galvanized steel protection by EHA was studied by electrochemical impedance spectroscopy. The Nyquist plots of electrodes treatment by different variants are shown in Figure 8. Based on these results, the equivalent scheme parameters and the characteristic values of the protective effect of EHA were calculated (Table 5).

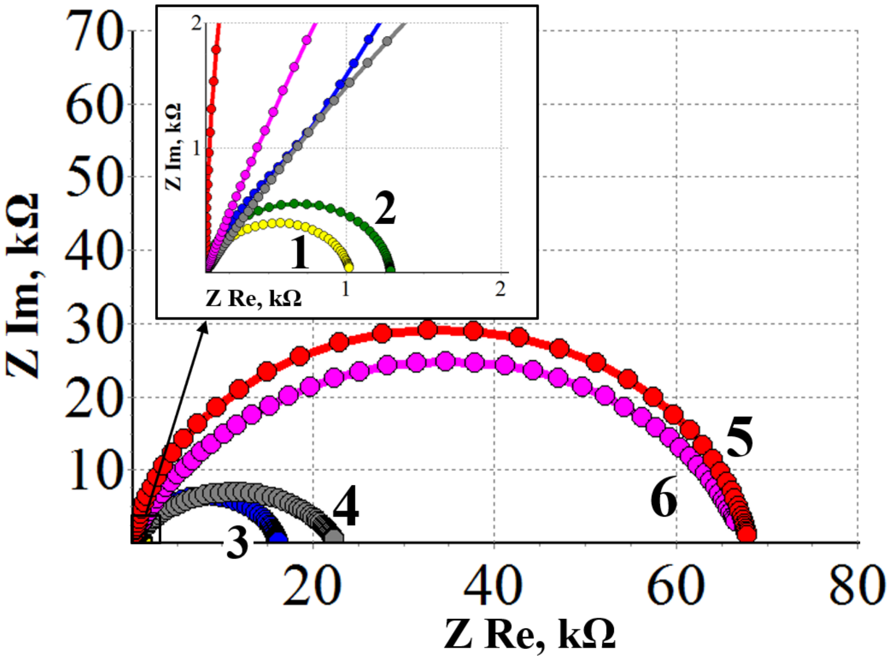

Figure 8. Nyquist plots of galvanized steel. Borate buffer, 1 mM, pH 7.36; t_{CT}=100°C, τ_{CT}=1 h. The numbering of experiments is identical to Figure 7.

Table 5. Characteristic values of the protective effect of EHA at different variants of galvanized steel treatment. The numbering of experiments is identical to Figure 7.

No.	Treatment conditions, corrosion inhibitor	γ_{bl}	γ_{act}	Θ, %	*Z*, %
2	Thermal treatment without inhibitors, $\tau_{exp}=24$ h	1.33	0.95	25	21
3	EHA, $\tau_{exp}=1$ h	23.5	0.68	96	94
4	EHA, $\tau_{exp}=5$ h	67.5	0.24	98	94
5	EHA, $\tau_{exp}=24$ h	323.5	0.08	99	96
6	EHA, $\tau_{exp}=48$ h	333.5	0.07	99	96

The surface oxide blocked about 25% of the surface upon thermal treatment of galvanized steel without corrosion inhibitors. The *Z* value after thermal oxidation of galvanized steel was 21%. The γ_{act} was close to 1, indicating a blocking mechanism of protection. Corrosion inhibition increased after chamber treatment of the electrode with EHA, as evidenced by the growth of the radii in the hodographs. The *Z* values increased up to 96%. *Z* values increased symbatically with τ_{exp} during the first day of exposure of the samples in air after chamber treatment. Longer exposure (larger τ_{exp}) was not accompanied by an increase in *Z*.

The values of Θ were close to 100% under the optimal conditions of chamber treatment of galvanized steel and $\tau_{exp} \geq 24$ h. Comparison of γ_{bl} and γ_{act} parameters indicates the blocking mechanism of corrosion inhibitor action. Moreover, the values of γ_{act} allow one to state that the corrosion is accelerated on the surface area that is not blocked by the inhibitor.

The results given above served as a basis for field tests that confirmed a high efficiency of EHA. Coating tarnishing was noticeable already at the first inspections on galvanized steel in the initial state and after treatment without inhibitors. Chamber treatment by EHA provided full protection of the metal for more than 2 years.

The “galvanized steel–NDA” system was further studied. The optimal conditions of chamber treatment determined in salt fog tests were 100°C and 1 h. The efficiency of galvanized steel protection by NDA under optimal conditions of chamber treatment is 120 h, *i.e.* it is higher than that by EHA (96 h).

Anodic polarization curves of galvanized steel upon treatment by different variants are presented in Figure 9.

The results of this series of experiments are similar to the results presented in Figure 7. NDA passivates galvanized steel and shifts its depassivation potential to the anodic region. The surface films are structurized during the first day of exposure in the air after chamber treatment, which results in an increase in their protective properties. The efficiency of NDA is commensurable with that of EHA.

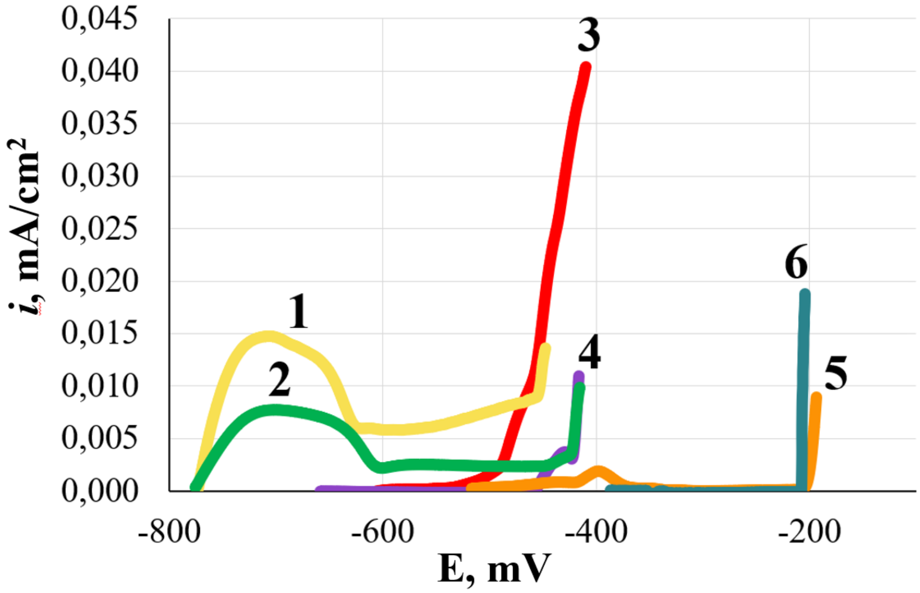

Figure 9. Anodic polarization curves of galvanized steel. Borate buffer, 1 mM, pH 7.36; $t_{CT}=100°C$, $\tau_{CT}=1$ h. 1 – samples in the initial state; 2 – samples after treatment without corrosion inhibitors, $\tau_{exp}=24$ h. Chamber treatment in NDA vapors at different τ_{exp}: 3 – 1 h; 4 – 5 h; 5 – 24 h; 6 – 48 h.

The impedance method was used at the next step of the study of the "galvanized steel–NDA" system. The Nyquist plots of galvanized steel after treatment by different variants are shown in Figure 10. The characteristic values of the protective effect of the corrosion inhibitor are given in Table 6.

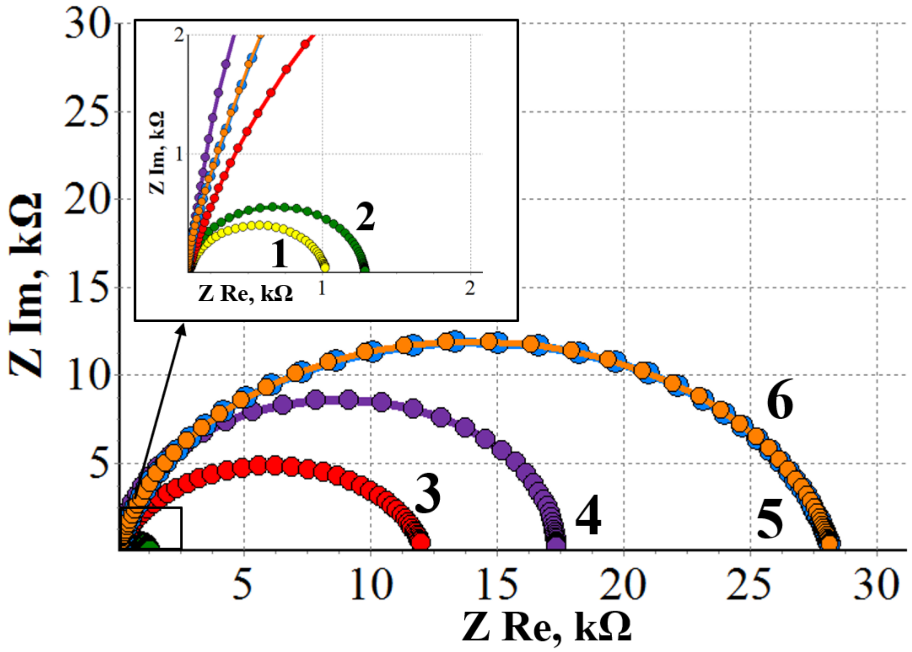

Figure 10. Nyquist plots of galvanized steel. Borate buffer, 1 mM, pH 7.36; $t_{CT}=100°C$, $\tau_{CT}=1$ h. The numbering of the experiments is identical to Figure 9.

Table 6. Characteristic values of the protective effect of NDA at different variants of galvanized steel treatment. The numbering of experiments is identical to Figure 9.

No.	Treatment conditions, corrosion inhibitor	γ_{bl}	γ_{act}	Θ, %	*Z*, %
2	Thermal treatment without inhibitors, τ_{exp}=24 h	1.33	0.95	25	21
3	NDA, τ_{exp}=1 h	12.67	1.06	92	93
4	NDA, τ_{exp}=5 h	19.47	0.94	95	94
5	NDA, τ_{exp}=24 h	15.13	2.19	93	97
6	NDA, τ_{exp}=48 h	15.87	2.08	94	97

Chamber treatment of the electrode with NDA resulted in corrosion inhibition. The degrees of metal protection *Z* were in the range from 93 to 97%. The *Z* value increased symbatically with the exposure time τ_{exp} during the first 24 h of sample exposure in the air after chamber treatment. Longer exposures (larger τ_{exp} values) did not increase the *Z* value. Under optimum conditions of metal treatment and at $\tau_{exp} \geq 24$ h, the degrees of surface coverage Θ were close to 100%. Comparison of γ_{bl} and γ_{act} indicates the blocking mechanism of NDA action.

Chamber treatment with NDA provided galvanized steel protection for more than 18 months under weathering conditions. The tests are still in progress, but it can already be concluded that NDA can be used for the temporary protection of metal items.

Chamber protection of "galvanic zinc–steel" contact pairs

The possibility of corrosion protection for "galvanic zinc–steel" contact couples was studied. The data on the effect of various compounds on the corrosion resistance of zinc were supplemented by the results of corrosion screening of the same compounds for carbon steel (Table 1).

Signs of corrosion on the samples in the initial state were observed on steel samples one hour after the beginning of tests under conditions of periodic moisture condensation. Thermal treatment does not affect the corrosion resistance of steel.

The compounds studied, excluding Captax, Altax and stearic acid, inhibited the initiation of corrosion. The largest protective effect (129 h) was noted for octadecylamine. For the other compounds, the protection time τ_{prot} did not exceed 24 h. Composite corrosion inhibitors demonstrated significant effects. Mixtures of stearic acid, linolenic acid, and EHA with urotropine provided steel protection for more than 500 h, while a mixture of EHA with octadecylamine, for 216 h. All the mixtures containing urotropine manifested a synergism of protective action ($\alpha \gg 1$). The components of the corrosion inhibitors containing no urotropine were antagonistic.

Obviously, the corrosion inhibitors for the "galvanic zinc–steel" contact couples should provide efficient protection of both zinc and carbon steel themselves. The compounds that inhibit corrosion initiation for both metals at least 200-fold were chosen for the study. These compounds include formulations of EHA with urotropine and EHA with octadecylamine.

The protective properties of these mixtures and their components with respect to "galvanic zinc–uncoated steel" contact couples were studied under conditions of periodic moisture condensation. The protection time τ_{prot} for both metals of the contact pair was determined separately (Table 7).

These data indicated a high protective capacity of both mixtures and served as a basis for the field tests. Mixtures of EHA with urotropine and EHA with octadecylamine provided full protection of contact couples in an urban atmosphere for 10 and 12 months, respectively. This is sufficient for the temporary protection of items comprising "galvanic zinc–steel" contact couples.

Table 7. Protective properties of adsorption films formed upon treatment of the "galvanic zinc–steel" contact couple. Conditions: periodic moisture condensation, $t_{CT} = 100°C$; $\tau_{CT} = 1$ h; $\tau_{exp} = 24$ h.

Treatment conditions	**τ_{prot}, h**	
	Zinc	**Steel**
Initial state	1.0	1.0
Thermal treatment without corrosion inhibitors	1.0	1.0
EHA with urotropine	168	1920
EHA with octadecylamine	1200	1920

Conclusions

1. α-Branched carboxylic acids, namely ethylhexane acid (EHA) and neodecanoic acid (NDA), provide efficient chamber protection of zinc.
2. The corrosion-electrochemical behavior of zinc and galvanized steel is similar but not identical. Nevertheless, the optimum temperature and duration of chamber treatment of these materials with EHA and NDA are the same: 100°C and 1 h, respectively.
3. Nanosized polymolecular adsorption films are formed on the metal surface under optimal conditions of chamber treatment of zinc and galvanized steel in EHA and NDA vapors; the films passivate the metals and stabilize their passive state in chloride-containing media. These films inhibit the thermal oxidation of zinc and are capable of structurization when electrodes are exposed in the air after chamber treatment for one day. The structurization is accompanied by a growth in the zinc protection efficiency.
4. Using the example of the "EHA–zinc" system, it has been found that the basic salt $C_4H_9–CH(C_2H_5)–COO–Zn–OH$ is formed on the metal in the course of chamber treatment. Dehydrogenation of this salt occurs upon the exposure in the air to give the

compound $C_4H_9{-}CH(C_2H_5){-}COO{-}Zn{-}O{-}Zn{-}OOC{-}CH(C_2H_5){-}C_4H_9$, which provides zinc protection.

5. Protection of zinc and galvanized steel from corrosion by EHA and NDA occurs by a blocking mechanism.
6. The protective films formed during chamber treatment of zinc with EHA and NDA are capable of self-healing in case of mechanical disruption of their integrity.
7. Mixtures of EHA with urotropine or octadecylamine provide efficient simultaneous protection of zinc and carbon steel.
8. Chamber treatment in EHA or NDA vapors can be used for temporary protection of zinc and galvanized steel items. It is expedient to use mixtures of EHA with octadecylamine or urotropine for temporary protection of "galvanic zinc–steel" contact couples.

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