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Influence of a nanometric organosilane film on the corrosion behaviour of zinc coating

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Zinc coatings treated by organosilane were investigated as environment-friendly replacements of the existing chromating processes. The aim was to compare the corrosion resistance of zinc coatings plated with organosilane, polyphosphate / organosilane with subsequent heat treatment at different temperatures. The corrosion resistance of organosilane coatings on zinc was examined by the neutral salt spray (NSS) method. Characterization of the organosilane coatings was performed by x-ray photoelectron spectroscopy (XPS). It has been found that zinc coating treatment with polyphosphate and subsequent treatment in organosilane with two hour heating at a temperature of 70 °C make it possible to achieve corrosion resistance close to that of chromated zinc coatings.

Key words: zinc, organosilane, phosphate, heating, corrosion, XPS

INTRODUCTION

In the last few years, the restrictions on chromate conversion coatings have been used for many years to protect metals from corrosion and improve the adhesion of paints and lacquers. They are highly effective, but unfortunately the Cr(VI) chemicals used in the conversion treatment are now to be carcinogenic [1]. The toxicity necessitates a search for alternative treatments. Due to regulations defined by the Europe Union, the use of chromate treatments was strongly restricted from 2007 [2].

In the last few years, the restrictions on the use of traditional pre-treatments and organic coatings for corrosion protection have been strictly limited due to both human health and environmental concerns.

The thresholds values for the dangerous and hazardous substances involved in the production and application of some most common pre-treatments and organic coatings of proved corrosion protection efficiency are becoming more and more stringent year by year. The need of alternatives to conventional protection systems promoted a huge number of studies and investigations aiming at the development of innovative and effective solutions with low environmental impact. By means of multifunctional silane hybrid molecules it is possible to design the final properties of pre-treatments and organic coatings.

In literature there is a tremendous amount of studies concerning the potential of organofunctional silane molecules as adhesion promoters between metallic substrates and organic coatings used for protection against corrosion phenomena [3–16].

Silanisation has been reported as an environmentally compliant alternative to replace the conventional chromate treatment [12, 14, 17–19].

The procedure is simple, being achieved by immersion of the metal substrate in alcohol or water based silane solutions for a short period. The final result is a stable thin silane film, possessing good coupling properties with organic polymers [20]. Silane films are 'passive' organic coatings, since they act essentially as a physical barrier which hinders

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the penetration of aggressive species towards the metallic substrate.

It is not enough to use a silane coating alone in order to substitute the chromate coating on zinc with a non-chromate one with equivalent protective properties, whereas the silane coating being only 100 nm thick does not provide sufficient Zn coating corrosion protection [21].

A more promising method for obtaining combined conversion coatings is to deposit a phosphate coating with its subsequent treatment with silicon organic compounds. By this means a conversion coating possessing properties characteristic of both polyphosphates and silanes could be formed on the Zn surface.

The purpose of this work was to study the corrosion resistance of Zn coatings treated with polyphosphates and silicon organic compounds.

EXPERIMENTAL

Steel St-10 specimens (50 × 50 × 1) mm in size were used for studies. They were plated with a 9 µm thick Zn coating in a weakly acidic zinc plating electrolyte according to the standard commercial electrolyte Likonda ZnSR (Zn). After that they were washed with deionized water, izopropile alcohol and dried in hot air. The traditional chromate conversion coating obtained in a solution called Likonda 22 was prepared for comparison.

To obtain phosphate coatings a condensed polyphosphate solution KF1 according to the patent [22] was used, by immersing a freshly zinc plated specimen in the KF1 solution for 30 sec at room temperature. Solution pH was adjusted to 1.3. After that the specimen was washed with deionized water and dried in hot air.

It is a common knowledge that polysilicone is distinguished for its good anticorrosion superhydrophobic and antifouling properties [23]. For example, the organosilicon compounds include ethyl silicate, propyl silicate, methyltrimethoxysilane, ethyltriethoxysilane, vinyltrimethoxysilane, etc. They may be used alone or in combination of two or more. To form silane coating we have chosen methyltriacetoxysilane (MTACS) from Gelest, Inc., USA (Fig. 1).

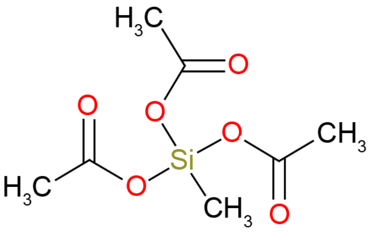

Fig. 1. Methyltriacetoxysilane

To obtain MTACS coatings Zn coatings were kept in an izopropile alcohol solution containing 10 g/l methyltriacetoxylane for 10 min under conditions described in [24]. After that the coatings were heat-treated for 2 h at 70 °C or for 0.5 h at 120 °C.

Neutral salt spray (NSS) testing was performed according to LST EN ISO 9227 [25]. Preliminary experiments have shown that the assessment of samples corrosion resistance until the occurrence of the first white Zn corrosion sites is not acceptable, because the NSS method is too strict to be used in our situation. Therefore it was decided to assess samples corrosion resistance according to LST EN ISO 7384 [26], that is to say, by the time until the first red local corrosion spots are produced on steel.

The organosilane coatings on various treated zinc surfaces were studied with an ESCALAB MKII spectrometer (VG Scientific, radiation Mg K_α – 1 253.6 eV, output 300 W). The pressure in the analytical chamber of the spectrometer was 1.33×10^{-8} Pa. The distribution of elements in the depth was determined by sputtering with an Ar^+ gun with the ion energy of about 2.0 keV. The samples were etched in the preparation chamber with the vacuum of $9.3 \cdot 10^{-3}$ Pa and the current of 20 µA; the duration of etching was 30 s. The maximum accuracy of the method was ±0.1 at.%. To reveal the composition of the obtained coatings, the photoelectron spectra of the C_{1s}, O_{1s}, N_{1s}, P_{1s}, P_{2p}, Zn_{2p}, Si_{2p} and the kinetic energy of Zn Auger (Zn $L_3M_{45}M_{45}$) were recorded. The empirical sensitivity factors for these elements were taken from the literature [27] and the spectra obtained were compared with the standard ones [28].

RESULTS AND DISCUSSION

Figure 2 presents a histogram of the corrosion resistance of Zn coatings treated either by the Likonda 22, KF1, MTACS or the combined method when Zn at first was treated with KF1, then with MTACS, as well as heated for 2 h at 70 °C or heated for 0.5 h at 120 °C.

It is seen that Zn treatment with the KF1 solution increases Zn coating corrosion resistance as compared to that of pure Zn (Fig. 2). These results agree with the data presented in the phosphating survey [29] that the phosphate coating enhances the corrosion resistance of metal. It is known [30] that the methoxy group ($O\text{-}CO\text{-}CH_3$) of the silane coupling agent [$CH_3\text{-}Si\text{-}(O\text{-}CO\text{-}CH_3)_3$] hydrolyzes in the presence of water-alcohol solution and forms silanols (Si–OH functions). These Si–OH functions prove to be very reactive in establishing the bond between the substrate material and organofunctional silanes [31]. The silanols are then expected to react with the naturally formed hydroxyl group on zinc or phosphate surfaces and form siloxane linkages or with each other to give a polysiloxane coating on the zinc or phosphate surface.

The mechanism by which the silane molecule interacts with the metallic substrate can be described as follows:

$$\begin{aligned} &R\text{–}Si(OCH_3) + OH\text{–}M \rightarrow \\ &\rightarrow R\text{–}Si(OCH_3)_2\text{–}O\text{–}M + CH_3OH. \end{aligned} \tag{1}$$

The organic chain R may carry substituent groups, such as the amino group, which can react with the oxide substrate leading to improved deposition of silane [32].

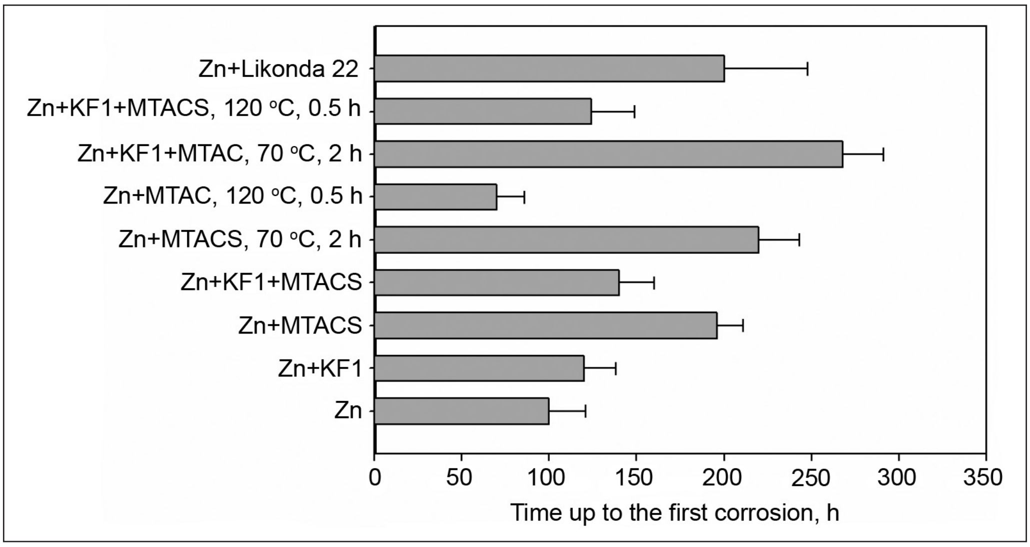

Fig. 2. Corrosion resistance of zinc coatings treated with Likonda 22, KF1, MTACS and KF1+MTACS by salt spray method. Columns – Fe corrosion beginning. The concentration of MTACS in izopropil alcohol is 10 g/l, 10 min. The specimens were heated for 2 h at 70 °C or for 0.5 h at 120 °C

The Zn coating treatment with MTACS as compared to the pure Zn coating increases corrosion resistance about two-fold Zn (Fig. 2). Literature data also support the fact that the silane coating is more resistant as compared to the phosphate coating [33]. For evaluation of galvannealed steel the salt spray test was performed, and 60 hours later it was possible to notice blistering and corrosion in the cut for the untreated and the phosphate treated systems. The silane system remained free of blistering and corrosion for at least 168 hours [34].

In the case of combined unheated coatings (Zn+KF1+MTACS), the corrosion resistance as compared to that of the pure Zn coating is nearly one and a half times higher (Fig. 2). However, in comparison with unheated Zn+MTACS coatings their corrosion resistance is about 25% lower. We suppose that one of the possible explanations could be different silane capacity to adhere to Zn more strongly as compared to phosphate. Literature data also testify that silane adheres to Zn more strongly as compared to gypsum ($CaSO_4$) [35].

The increase in corrosion resistance of Zn+MTACS and Zn+KF1+MTACS coatings heated at 70 °C for 2 hours (Fig. 2) can be ascribed to dehydratation of KF1 coatings and to formation of stable and homogeneous MTACS coatings.

The corrosion resistance of Zn+MTACS coatings heated at 70 °C for 2 hours, as compared to that of unheated Zn+MTACS coatings, has a tendency to increase (Fig. 2). It is known [36] that the drying of silane promotes silane condensation on the substrate surface and removes some of the loosely absorbed molecules probably by simple evaporation. According to Fousse [37], heating the organic film on the metal substrate may help it react with metal and with itself, and may enhance the cross-linking through physical entanglement. Heating also helps the evaporation of the solvents. All these effects contributed to the compactness of the organic film and the improvement of its hydrophobicity.

Corrosion resistance of Zn+KF1+MTACS coatings heated at 70 °C for 2 hours is nearly twofold higher as compared to that of Zn+KF1+MTACS coatings without heating and reaches about 270 hours (Fig. 2). For comparison purposes it should be said that the chromated Zn coating under analogous conditions provides corrosion resistance for 200 hours and has a good agreement with data [38].

Zn+MTACS and Zn+KF1+MTACS coatings heating at 120 °C for 0.5 hour negatively affects their corrosion resistance. The corrosion resistance of Zn+MTACS, as compared to that of Zn+MTACS unheated coatings, decreases approximately twofold (Fig. 2).

The XPS results obtained for the Zn sample pre-treated with the silane coating show that both heated and non-heated samples of the surface film are composed of ZnO and silane (Table 1). Metallic Zn is also observed in Zn ($L_3M_{45}M_{45}$) spectra indicating non-uniformities in the silane film depth. From binding energy (BE) changes called chemical shifts (binding energy shifts) it can be determined what compounds the studied element forms depending on its chemical state and the environment. The core-level shifts could be attributed to changes in the electronegativities (Pauling term), the ionicities (Madelung term), and the final states (relaxation term) in the environment of the photoionized atom [39]. Zn is less electronegative than Si (Pauling values, Zn: 1.6, Si: 1.9), which suggests a greater ionic character for ZnO. These analyses indicated that the core-level shifts of both Zn and Si were due to the formation of a Si–O–Zn bond. This is consistent with the values reported in existing literature [40].

The data in Table 1 suggest that on the surface of the Zn+MTACS specimen heated at a temperature of 70 °C for 2 hours the quantities of Zn, O and Si decrease, while the

Table 1. **Elemental composition (in at.%) of zinc coatings (Zn), phosphated zinc (Zn+KF1) coatings and phosphated zinc treated with methytrilacetoxysilane (Zn+KF1+MTACS) coatings obtained by using binding energy, Zn Auger kinetic energy**

Samples	Zn, at.%; Binding energy, eV	Zn ($L_3M_{45}M_{45}$) Kinetic energy, eV	O, at.%; Binding energy, eV	Si, at.%; Binding energy, eV	C, at.%; Binding energy, eV	P, at.%; Binding energy, eV
Zn	26.7; 1 021.6	988.1; 990.3	52.7; 531.1	–; –	20.6; 284.5	–; –
Zn+KF1	3.9; 1 021.4	987.7; 990.1	53.0; 530.6	–; –	29.4; 284.5	11.9; 133.1
Zn+MTACS	16.7; 1 021.5	988.0; 991.1	47.2; 531.2	2.4; 102.3	33.7; 284.5	–; –
Zn+MTACS, 2 h, 70 °C	13.6; 1 021.5	988.1; 991.1	43.7; 531.2	1.8; 102.4	41.0; 284.5	–; –
Zn+MTACS, 0.5 h, 120 °C	20.1; 1 021.7	988.3; 991.3	41.9; 530.2	2.9; 102.1	35.1; 284.5	–; –
Zn+KF1+MTACS	8.5; 1 022.2	987.2; 989.6	51.5; 531.6	2.75; 102.5	26.2; 284.5	11.0; 133.8
Zn+KF1+MTACS, 2 h, 70 °C	4.4; 1 022.0	987.2; 989.6	44.5; 531.4	2.8; 102.7	38.0; 284.5	10.3; 133.6
Zn+KF1+MTACS, 0.5 h, 120 °C	6.9; 1 022.2	987.3; 989.5	45.3; 531.5	4.14; 102.4	35.4; 284.5	8.2; 133.6

Table 2. **Elemental composition (in at.%) of zinc coatings (Zn), phosphated zinc (Zn+KF1) coatings and phosphated zinc treated with methytrilacetoxysilane (Zn+KF1+MTACS) coatings obtained by using binding energy, Zn Auger kinetic energy after 2.5 nm of the coatings were etched**

Samples	Zn, at.%; Binding energy, eV	Zn ($L_3M_{45}M_{45}$) Kinetic energy, eV	O, at.%; Binding energy, eV	Si, at.%; Binding energy, eV	C, at.%; Binding energy, eV	P, at.%; Binding energy, eV
Zn	72.5; 1 021.3	988.1; 992.8	26.4; 530.1	–; –	1.15; 284.5	–; –
Zn+KF1	18.6; 1 022.6	986.8; 989.2	59.6; 531.8	–; –	6.96; 284.5	14.3; 134.1
Zn+MTACS	46.0; 1 021.8	988.2; 991.1	45.7; 530.3	–; –	8.4; 284.5	–; –
Zn+MTACS, 2 h, 70 °C	39.1; 1 021.5	988.2; 991.2	43.3; 530.0	–; –	17.5; 284.5	–; –
Zn+MTACS, 0.5 h, 120 °C	46.1; 1 021.7	988.4; 991.2	45.0; 530.1	0.91; 102.1	8.0; 284.5	–; –
Zn+KF1+MTACS	25.0; 1 022.1	987.5; 989.5	59.3; 531.2	–; –	2.2; 284.5	13.5; 133.5
Zn+KF1+MTACS, 2 h, 70 °C	18.1; 1 022.1	987.5; 989.5	58.6; 531.2	–; –	8.96; 284.5	14.3; 133.6
Zn+KF1+MTACS, 0.5 h, 120 °C	19.5; 1 022.4	987.2; 989.2	57.7; 531.6	1.55; 102.8	7.8; 284.5	13.5; 133.8

quantity of C increases, as compared to those of the specimen at 20 °C. At 70 °C a further formation of the silane coating supposedly occurs giving a covalently bonded, crosslinked polysiloxane layer on the zinc surface which improves its hydrophobicity [37]. The BE value of Si_{2p} was found to be 102.4 eV, which is coincident with silicon BE in methyltriacetoxysilane [41] or zinc silicate [27]. When the heating temperature was increased to 120 °C, the quantity of Zn and Si on the surface of silane coating increases, while the quantity of O decreases. The O_{1s} XPS spectra exhibited an asymmetric peak, indicating two different bonds of oxygen 530.2 and 531.4 eV, which corresponded to ZnO and silane, respectively [39]. The Si_{2p} binding energy of the Zn+MTACS specimen heated for 0.5 h at 120 °C diminishes to 102.1 ± 0.1 eV and becomes close to the BE of the nanocrystalline samples, which implies a lesser polymerisation [42]. Thus at a heating temperature of the Zn+MTACS specimen of 120 °C, the silane coating cracks due to formation of zinc silicate and therefore an increased amount of Si and a decreased amount of C were detected in the coating.

It was found that the inclusion of a post-treatment heating procedure in the process significantly increases the polarization resistances of the coatings which means a better protection of zinc surfaces based on the deposition of silicates from sodium silicate solutions [43].

The Zn Auger lines values (Table 1) show that on the surface of Zn+MTACS coatings heated at 120 °C higly oxidized (Zn ($L_3M_{45}M_{45}$) $\rightarrow$ 988.0–988.3 eV) is present in the form of ZnO and partially in the metallic state (991.1–991.3 eV) [26]. However, the state of the Zn+MTACS surface does not change keeping it at 20 and 70 °C.

The Auger peaks 987.2–987.3 eV obtained for the Zn+KF1+MTACS samples show that zinc silicate (similar to hemimorphite) or $Zn(OH)_2$ is present on the surface. Therefore it can be stated that we have silicon covalent binding through oxygen with zinc (Si–O–Zn bond).

After etching with Ar^+ ions of 2.5 nm of the Zn specimen coated with silane, Si was not detected in any specimen, with the exception of that heated at 120 °C (Table 2). This suggests that the silane coating is very thin.

The data in Table 1 show that the sum of the quantities of Si and P is a constant value of approximately 13.5 at.% independent of thermal treatment. The same quantity of P was detected after 2.5 nm of the specimen were eched (Table 2), when silane is practically missed. Most probably Si organic compound is adhered to the polyphosphate coating. An increase in the quantity of carbon on the surface of the sample heated at 70 °C shows that, as for the unphosphated zinc, methytrilacetoxysilane making a bond with phosphorus is outward-directed by its aliphatic part.

After polyphosphate coating treatment with silane an increase in phosphorus BE P_{2p} from 133.1 eV to 133.8 eV is observed (Table 1). However, after heat treatment of this sample both at 70 °C and 120 °C temperatures the BE slightly decreases to 133.6 eV. Most probably, a P and Si organic compound derivative is formed. The BE of Si_{2p} increases from 102.3 eV to 102.7 eV with an increase in sample treatment temperature from 20 °C to 70 °C, though the increase is only slight (Table 1).

Thus, XPS studies have shown that Zn+MTACS coatings heated at 120 °C for 0.5 h lose their corrosion resistance because of the degradation of the silane coating (the latter turns into zinc silicate). Meanwhile, the corrosion resistance loss of Zn+KF1+MTACS coatings heated at 120 °C for 0.5 h can be explained by MTACS coating degradation and KF1 coating corrosion resistance increase of which is under the former coating because of higher dehydration of the phosphate coating [44].

The fact that phosphate coatings retained their corrosion resistance at certain treating temperatures is in good agreement with the data reported by other researchers [45], who demonstrated that the corrosion properties of phosphate coatings do not change up to 100 °C (or even enhance), while at 200 °C they are less by nearly half. Inasmuch as the coatings Zn+KF1+MTACS treatment in our case was performed at a temperature of 120 °C, therefore no essential changes in the phosphate coating were likely to happen. Thus the corrosion resistance of Zn+KF1+MTACS coatings heat treated at 120 °C for 0.5 h is close to that of non-heated Zn+KF1+MTACS coatings.

CONCLUSIONS

Salt spray tests showed the improved corrosion resistance of the Zn+KF1+MTACS coatings heated at 70 °C for 2 hours. Zn coatings treatment with the polyphosphate solution (KF1) and organic silane (methyltriacetoxysilane) with subsequent treatment at 70 °C for 2 h makes it posible to achieve corrosion resistance values close to those of chromated zinc. From XPS data we can conclude that heating of methyltriacetoxysilane coatings at 120 °C for 0.5 h destroys them because of the formation of zinc silicate and dehydration of the constituent phases of the phosphate crystals occurs.

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NANOMETRINIO SILICIO ORGANINIO JUNGINIO PLĖVELĖS ĮTAKA CINKO DANGOS KOROZINEI ELGSENAI

Santrauka

Cinko dangos, apdorotos silicio organiniu junginiu, buvo tirtos kaip galimas ekologiškas pakaitalas esamiems chromatavimo procesams. Darbo tikslas – palyginti cinko dangų, padengtų silicio organiniu junginiu, polifosfatu / silicio organiniu junginiu su vėlesniu terminiu apdorojimu, korozinį atsparumą skirtingose temperatūrose. Korozinis atsparumas tirtas taikant neutralaus druskos rūko (NSS) metodą. Silicio organinio junginio dangų charakteristikos nustatytos panaudojus rentgeno fotoelektroninę spektroskopiją (XPS). Nustatyta, kad cinko dangų apdorojimas polifosfatu, po to silicio organiniu junginiu ir 2 val. kaitinimas 70 °C temperatūroje leidžia pasiekti korozinį atsparumą, artimą chromatuotoms cinko dangoms.