

## Investigation on the corrosion protection ability of nanocomposite hybrid coatings

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The electrochemical behaviour of sol-gel prepared, nanocomposite coatings was investigated with respect to proving their corrosion protective capabilities. Amorphous ZrO<sub>2</sub> (5 nm) and crystalline CeO<sub>2</sub> (10 nm) nanoparticles were embedded in the sol-gel matrices with solid content ranging 8% to 20%. The sol-gel coatings were applied on AA 2024 substrate. Additionally, a part of these sol-gel-coated samples was covered as an additional protection with two layers of an industrial paint system provided by the European Aeronautic Defence and Space Company (EADS). The thickness of the prepared single sol-gel coatings, and the top-coated samples varied between 13 to 19 μm, and from 40 to 78 μm, respectively. The corrosion protection performance of the obtained samples was determined by means of electrochemical measurements, including voltammetry and Electrochemical Impedance Spectroscopy (EIS).

**Key words:** nanocomposite, hybrid coatings, corrosion protection, aluminium alloy 2024, electrochemical tests.

### INTRODUCTION

Aluminium is an important metal in industry owing to its specific characteristics. Thus, it is widely used as a material for automobiles, aviation, household appliances, containers, and electronic devices. Despite its apparent advantages, in a number of cases, however, the use of aluminium is limited by corrosion in aggressive environments especially those containing chloride ions [1].

It is known that copper as alloying element particularly increases Al corrosion by forming galvanic couples. For this reason, aluminium alloys with high copper content, for example AA2024, corrode more easily than those with a lower copper content. The corrosion rate depends on the alloy composition and the local environment. Today, corrosion protection of light-weight aluminium alloys is achieved by ecologically harmful wet chemical processes like chromating or phosphating. It is intended to replace Cr(VI), which is known to be dangerous to human health and environment [2].

In the industrial practice, the metallic substrates are pre-treated before application of an organic paint in order to improve the adhesion between the polymer and the metal, and to provide additional long-term corrosion protection. So far, the most often used pre-treatments contain chromates as

active agents. However, chromate compounds are strongly carcinogenic [3].

Due to the prohibited use of chromium compounds in corrosion protection, a lot of scientific effort is invested into development of new corrosion protection species [4–6]. One promising approach is the work of Mansfeld [7], who proposed to coat aluminium sheets with aqueous solutions of cerium, and molybdenum salts. Since the treatment of the aluminium sheets takes several hours, an industrial applicability appears questionable.

In previous works [8], the corrosion protection capabilities of hybrid inorganic/organic sol-gel coatings with incorporated zirconia or ceria nanoparticles [3, 9–11], and even with mixtures of them [12] were evaluated. In the present case, the process time interval for the sample sheets is much shorter, so the treatment presented here is considered to be more attractive for industrial use.

The general purpose of the present work is to investigate the corrosion protection ability of nanocomposite hybrid coatings by comparative measurements based on the principles of the durability tests. These were performed by both Electrochemical Impedance Spectroscopy and Linear Sweep Voltammetry methods. By repeating these measurements after certain periods of time, it was possible to examine their evolution with the residence time of the coatings in the corrosive medium. Hence, it was possible to define the changes of the polarization resistance with the time.

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## EXPERIMENTAL

### *Sol-gel preparation*

In order to synthesize the sol-gel-material, three different solutions were prepared separately. For obtaining the first solution the organic substructure of a hybrid material, consisting of 2,2'-bis-(4-hydroxyphenyl)-propane (BPA), was dissolved in an organic solvent. The second solution was obtained by hydrolysis of a mixture of tetraethyl orthosilicate (TEOS), methyltriethyl orthosilicate (MTEOS) and addition of SiO<sub>2</sub> nanoparticles, as components of the inorganic network, catalysed by concentrated hydrochloric acid (HCl). In order to create a chemical cross-linking between the organic substructure and the inorganic network, 3-glycidoxypropyltrimethoxysilane (GPTS) was used. To assist the hydrolysis, 0.1 M hydrochloric acid was added as a catalyst. CeO<sub>2</sub> nanoparticles (Aldrich) or ZrO<sub>2</sub> nanoparticles [9<sup>7</sup>], respectively, were added to the solution at concentrations of up to 16% in the case of ZrO<sub>2</sub> and 20% in the case of CeO<sub>2</sub>, calculated with respect to the solid material.

After the completion of the hydrolysis, all the three solutions were mixed together. Prior to the coating process, 1-methylimidazole was added to the mixture to start the organic polymerization; the detailed procedure has been described elsewhere by Schmidt *et al.* [10]. These sol-gel coatings were synthesized at the Leibniz Institut für Neue Materialien GmbH, Saarbrücken, Germany, using the above described technology.

In order to examine synergistic effects of the sol-gel-coating in combination with an industrial coating system, the sol-gel-material was applied and cured. Afterwards a thin layer of pyrolytic silica was applied to improve the adhesion of the commercially available primer and top-coat paint system, for additional protection. Two-component epoxy-based paint system was supplied by Mankiewicz, Hamburg.

### *Surface treatment, coating deposition and curing procedure*

Aircraft Al-alloy AA 2024 was used for the deposition of the sol-gel coatings. The aluminium sheets were degreased with acetone, cleaned with Metaclean T2001 (Chemie Vertrieb, Hannover, Germany), and subsequently etched in an alkaline cleaner (Turco Liquid Aluminetch Nr. 2 supplied by Turco Chemie, Germany). Afterwards, the substrates were desmutted by Turco Liquid Smutgo NC. The sol-gel-coatings were applied by dip-coating process, and cured at 120°C for 4 h. One set

of samples was cured only for one hour in order to examine the influence of a shorter curing time interval.

After the curing step, one part of the samples was coated, using an industrial paint system, applied by spray coating deposition. The primer layer was applied, and the samples were left at room temperature for 24 hours. After that, the topcoat layer was applied. The samples were dried again at room temperature for at least 24 hours prior to the electrochemical investigations. As a result, these samples present four layers coating system, as it is shown in Figure 1.

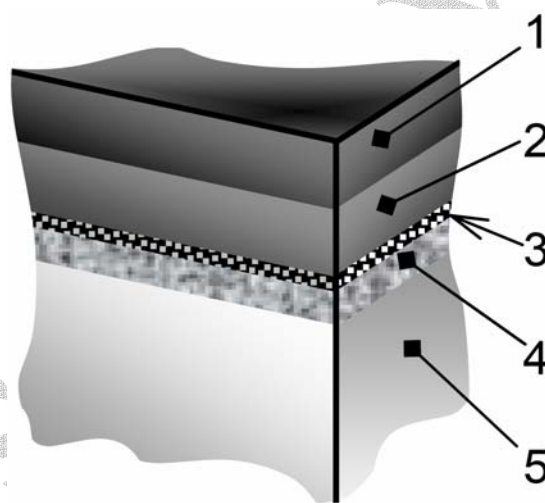


Fig. 1. Schematic view of the ordering of the applied four layers coating system.

- 1- EADS top-coat (thickness 20-30microns);
- 2- EADS primer coat (thickness 20-30 microns);
- 3- SiO<sub>2</sub> layer (50-100 nm);
- 4- Sol-gel coating (5-13 microns);
- 5- coated metal substrate.

A brief description of the samples, which are object of the present study, is given in Table 1.

### *Evaluation of corrosion protection ability by electrochemical examination*

For the purpose of the measurements a three-electrode cell was used. Its construction is depicted in Figure 2. The measured samples (WE) were used as working electrodes. A part of their surface was exposed with the form of circus, whose area was equal to 1.93 cm<sup>2</sup>. The counter electrode (CE) represented a platinum net with highly developed area. The reference electrode (RE) was a standard one Ag, AgCl/3M KCl, produced by METROHM. The cell was connected with a potentiostat/galvanostat PG30/2, product of AUTOLAB. The impedance spectra were extracted by the additive module – Frequency Response Analyzer (FRA-2), of the same company.

**Table 1.** Characteristics of the coatings of research.

T1	Number of the sample	Name of the sample	Constitution of nanoparticle's containing layer	Curing time at 120°C, h	Total number of the layers	Thickness of nano-particle's containing layer, $\mu\text{m}$	Measured thickness of the total coating $\bar{X}_{10}$ , $\mu\text{m}$
1	P-1	AA2024	-	-	-	-	-
2	P-2	Ref EADS**	-	-	2	-	$34.5 \pm 0.8$
3	P-3	Hydrophil + CeO <sub>2</sub>	Epoxyasilane; Aluminosilan with CeO <sub>2</sub> 8.6%	4	1	5–7	$7.3 \pm 0.3$
4	P-4	Hydrophil-CeO <sub>2</sub> EADS,	Epoxyasilane; Aluminosilan with CeO <sub>2</sub> 8.6%	4	4	5–7	$60.8 \pm 0.6$
5	P-5	GMT 8% ZrO <sub>2</sub> 1h	Ethoxysilane, methyltriethoxysilane Tetraethoxysilane ZrO <sub>2</sub> 8%	1	1	5–7	-
6	P-6	GMT 8% ZrO <sub>2</sub> 1h EADS	Ethoxysilane, methyltriethoxysilane Tetraethoxysilane ZrO <sub>2</sub> 8%	1	4	5–7	-
7	P-7	GMT 8% ZrO <sub>2</sub> 4h	Ethoxysilane, methyltriethoxysilane Tetraethoxysilane ZrO <sub>2</sub> 8%	4	1	5–7	$5.6 \pm 0.3$
8	P-8	GMT 8% ZrO <sub>2</sub> 4h EADS	Ethoxysilane, methyltriethoxysilane Tetraethoxysilane ZrO <sub>2</sub> 8%	4	4	5–7	$73 \pm 0.7$
9	P-9	GMT 16% ZrO <sub>2</sub> 4h	Ethoxysilane, methyltriethoxysilane Tetraethoxysilane ZrO <sub>2</sub> 16%	4	1	10–13	$13.1 \pm 1.0$
10	P-10	GMT 16% ZrO <sub>2</sub> 4h EADS	Ethoxysilane, methyltriethoxysilane Tetraethoxysilane ZrO <sub>2</sub> 16%	4	4	10–13	$75.1 \pm 1.4$
11	P-11	GMT 20% CeO <sub>2</sub> 4h	Ethoxysilane, methyltriethoxysilane Tetraethoxysilane ZrO <sub>2</sub> 20%	4	1	7–9	$7.1 \pm 0.6$
12	P-12	GMT 20% CeO <sub>2</sub> 4h EADS	Ethoxysilane, methyltriethoxysilane Tetraethoxysilane CeO <sub>2</sub> 20%	4	4	7–9	$74.9 \pm 1.9$

GMT – Gel Matrix; \*\* EADS – European Aerospace Defense and Space Company.

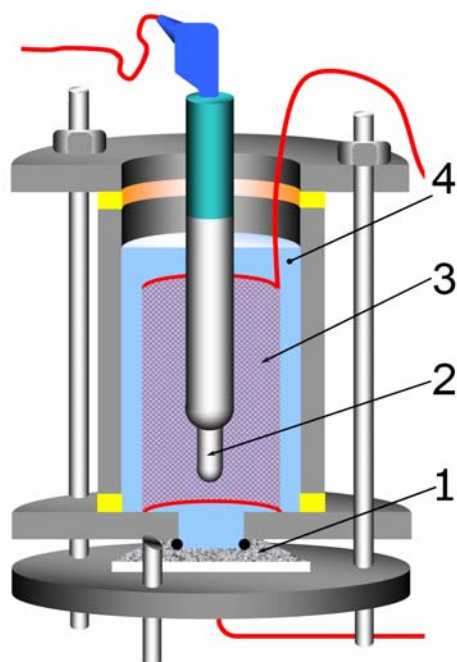


Fig. 2. Schematic view of three electrode cell.

1 – working electrode (which is also the sample),  
 2 – reference electrode, 3 – Winkler's counter electrode,  
 (represented by platinum net), 4 – electrolyte (liquid corrosive medium).

The average value of the coating's thickness was measured in ten different points of the area selected

for exposure. The measurements were performed using POSITECTOR 6000 FNS2 device, produced by DeFlesco corp. USA, before their passing through the electrochemical tests. The obtained values are represented in Table 1.

- The part of the sample's surface without visual defects was selected for exposure.

- The cell was not dismantled until the end of the measurements, and all of the measurements passed without removing or replacing the samples. By this manner, one of the basic error sources were avoided. The first measuring cycle with each sample was executed four hours after the immersion of the sample in the electrolyte.

- The polarization curves were obtained under conditions of potentiodynamic regime. It was performed within relatively short potential interval, which always includes the Open Circuit Potential (OCP). That potential interval was varied from (OCP -0.15V) to (OCP +0.40V), while OCP value was measured immediately before every measuring cycle. Thus a more pronounced polarization, which might cause undesired damages to the coatings, was avoided. By this manner, the difference between OCP and Corrosion Potential ( $E_{\text{corr}}$ ) was reduced, where the latter is defined based on the complete polarization curve.

- The potential's sweep rate was 1 mV/s. Thus, by that slow decrement, results with satisfying reproducibility were obtained, in spite of inability to avoid completely the influence of the capacitive component of the current.

- The impedance spectra were obtained at open circuit potential and frequency's interval from  $1 \times 10^6$  to  $1 \times 10^{-2}$  Hz. The amplitude of excitation signal was equal to 10mV in this way the linearity of the voltage-amperage dependence is achievable. So, the impedance spectrum corresponds to the most important requirements, described in the literature [12]: linearity of the response signal; stability of the system in the course of every one measuring cycle; relationship between the input and output electric signals, without any kind of perturbations.

All of the experiments have been accomplished in liquid media, consisting of 3% solution of NaCl in distilled water.

## RESULTS AND DISCUSSION

### *Voltammetric measurements*

For corrosion protection by applying of multilayer coatings on the metal surface, every layer has specific destiny. To clarify the contribution of every layer for the total corrosion protection, comparative systematic researches were carried out. They were related to the measurements of the following parameters: Open Circuit Potential (OCP), corrosion current's density ( $I_{corr}$ ) and the polarization resistance ( $R_p$ ), respectively. The measurements were carried out over monolayer sol-gel coatings, the polymer EADS coating, and combination of these basic layers.

The respective OCP and the development with the exposure time for both monolayer and multilayer coatings are represented in Figure 3. For comparison - development of the values of OCP with the exposure time for the aluminium alloy is also represented.

From that figure, it is observable that in all the curves OCP shifts to more positive values, due to the application of layers over the metal surface.

In the case of polymer coating of EADS, that shift is consequence of blocking of the metal surface, which is the reason of the simultaneous decrease in the rates of both partial reactions: the cathodic (oxygen reduction), and the anodic (ionization of the metal) side. The shift mechanism in the case of the sol-gel coating is different. The  $CeO_2$  and  $ZrO_2$  nanoparticles incorporated in the coating, act as of inhibitors. [13, 14].

The polarization resistance ( $R_p$ ), as a criterion for the protective ability of the coatings, was deter-

mined from the shape of the polarization curves by use of GPES software, and the corrosion current's density was defined from the intersection point of the slopes of the same curves.

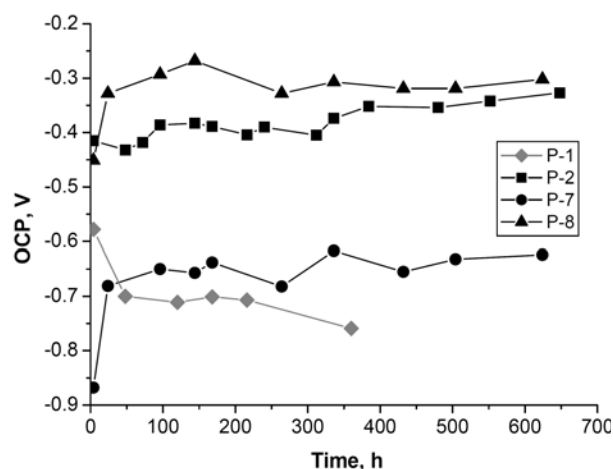


Fig. 3. Development of OCP with the exposure time. (P-1) - the aluminium alloy; (P-2) – the polymer EADS coating; (P-7) - the sol-gel monolayer coating; (P-8) – multilayer coating, consisted on both of EADS, and sol-gel layers.

Figure 4 represents the polarization curves for: sol-gel coating (a), EADS polymer coating (b), and combination of them (c), after 96 hour of exposure to the corrosive medium.

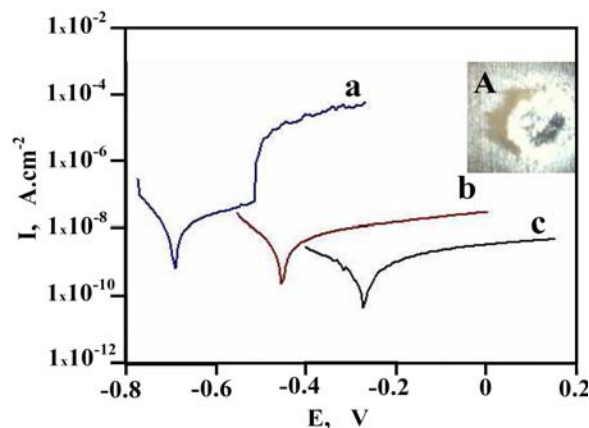


Fig. 4. Polarization curves of sol-gel coating (a), EADS polymer coating (b), and combination of them (c), after 96 hour of exposure in the corrosive medium.

The polarization curves of all the sol-gel coatings have shown a sharp jump of the current at definite value of the potential, which belongs to the anodic branch of the curve (Fig. 2a). At that potential, the electrode becomes not polarizing, because of the appearance of pitting on the sample's surface (see the metallographic photography in Figure 4 – magnification 232 times). No pitting corrosion is observable in the case of the presence of EADS upper layers. The pitting appearance shows that the monolayer

sol-gel coatings cannot be used as effective protective layers, without addition of polymer. So, the sol-gel inner layer possesses the function of “reservoir” of passivating particles of CeO<sub>2</sub> or ZrO<sub>2</sub>.

The time development of the corrosion current density for each one coating was followed. The obtained experimental values have not shown satisfying reproducibility. In order to “catch” the tendency of the protective abilities of every one of the coatings, we have accepted that, in the present case, the use of average values is a reasonable approach. These values were calculated by the individual values of the current for every single measurement. The limits of the time interval, for which the corresponding values were calculated, were selected to be between 96 and 624 hours. The reason for the selection of that interval was that the experience shows that if the exposure time is shorter than 96 hours, the multilayer coating has behavior of a dielectric: only weak current oscillations are observable, independent on the electrode’s potential. It is necessary to marked that after prolonged exposure to the corrosive medium it becomes possible that measurable Faraday’s currents appear, which are dependable on the electrode’s potential, and the obtaining of a polarization curve becomes possible. That behaviour of the multilayer coatings could be explained by gradual penetration of electrolyte in the micro-pores of the coatings. The average values of the corrosion current density, calculated for all investigated samples are represented on Table 2. For a better understanding, these results are graphically represented on the diagram of the Figure 5.

Following the data in Table 2, and the diagram in Figure 5, the following conclusions could extracted:

- The polymer EADS coating, accepted by us as reference, divides conditionally the rest of the coatings, into two basic groups, from the point of view of their corrosion current density, and respectively - their protective abilities, because all the monolayer sol-gel coatings have lower protective ability than the multilayer.

- The corrosion current decreases five times with the increase of the content of the ZrO<sub>2</sub> in the sol-gel inner layer, from 8% to 16% (see P-7 and P-9). Because the thickness of the corresponding coatings is not exactly identical, that conclusion can not be accepted absolutely as a decisive one.

- The coatings containing ZrO<sub>2</sub> possess better protective ability than those with CeO<sub>2</sub>, for example see P-9 and P-11.

- Upon increasing the curing time from 1 to 4 hours, (P-5 and P-7), a slight decrease of the protective ability of the coatings is observable.

**Table 2.** Average values of the current density (AVCD) for all of the coatings (P-1 to P-12) object of the present study.

Coating	AVCD, A/cm <sup>2</sup>
P-1	1.5×10 <sup>-6</sup>
P-2	9.3×10 <sup>-10</sup>
P-3	1.9×10 <sup>-8</sup>
P-4	6.6×10 <sup>-10</sup>
P-5	6.0×10 <sup>-9</sup>
P-6	1.2×10 <sup>-10</sup>
P-7	8.8×10 <sup>-9</sup>
P-8	7.8×10 <sup>-11</sup>
P-9	1.7×10 <sup>-9</sup>
P10	1.7×10 <sup>-10</sup>
P-11	5.3×10 <sup>-9</sup>
P-12	5.3×10 <sup>-10</sup>

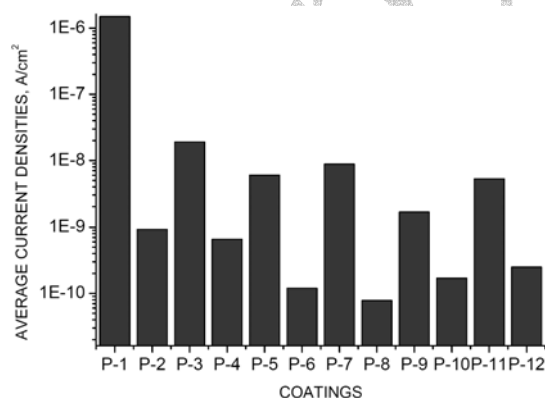


Fig. 5. Histogram of AVCD values vs. the number of sample.

### Impedance measurements

For quantitative evaluation of that kind of measurements, one typical value for EIS is the impedance module  $|Z|$ , determined at frequency 10 mHz [15]. The  $|Z|$  value has the character of total alternative current’s resistance, and it is defined by the following relationship:

$$|Z| = \sqrt{(\text{Re} Z)^2 + (\text{Im} Z)^2}, \quad (1)$$

where ReZ is the real component and ImZ – the imaginary component of the impedance, measured at certain frequency. It is well known, that the coatings with higher protective ability possess higher  $|Z|$  value

The experimental data obtained from the impedance measurements are represented in Bode coordinates. For the evaluation of corrosion, these coordinates are more preferable than the Nyquist coordinates commonly used in the electrochemical kinetic researches. The Bode coordinates are more expressive and the values for the impedance and phase shift have equal relative weight for every frequency decade. For the purpose of the present study, this

kind of coordinates is also more convenient from other point of view: The data measured with the multilayer coatings, represented in Nyquist coordinates, have shown significant dissipation of the experimental points. That dissipation is not so expressed and it does not change the dependence of

$|Z| - \varphi$  on the frequency, when they are represented in Bode coordinates.

The spectra of one monolayer (P-7), one multilayer (P-8), and the reference coatings (P-2), and their development with the time are represented in Figure 6.

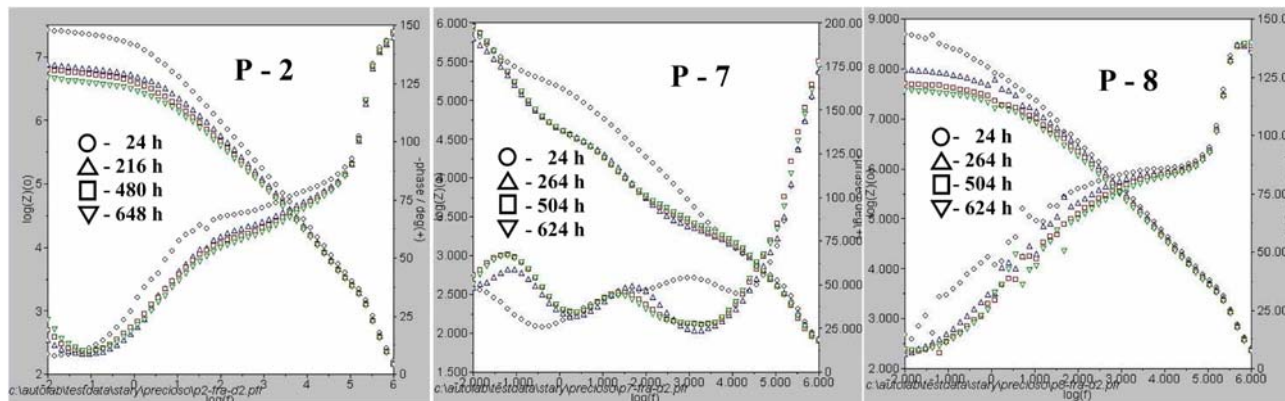


Fig. 6. Development of the impedance spectra at exposition of coatings P-2, P-7, P-8 in the corrosion medium.

The spectra of the coatings with EADS top coat are identical with one another, and they almost do not change with the exposure time. At a higher frequency range of these spectra, one not clearly shaped maximum appears in the relationship between the phase shift and the frequency. It means that there is one slightly expressed time constant, which is probably related to the polymer layer's capacitance and the ohmic resistance of its pores, but not to any corrosion processes on the phase boundary between the metal and the coating could be accepted. This kind of coatings ensures promising and durable corrosion protection of the substrate.

A large variety of the shape of the impedance spectra is observable in the case of monolayer sol-gel coatings. In their case, upon exposure in a corrosive medium, changes of entire impedance spectra appear. These changes are more remarkable at the low frequency range of the spectra. There are more than one clearly expressed maxima in the relationship between the phase shift and the frequency. The time constants corresponding to them are related to transport and electrochemical (corrosion) processes, on the metal/coating boundary. This kind of coatings does not supply durable promising protection of the substrate. The only exception exclusion is the coating P-9, which contains 16%  $ZrO_2$ . Its impedance spectra are similar to those of the multilayer coatings, although the coating is without a top coat.

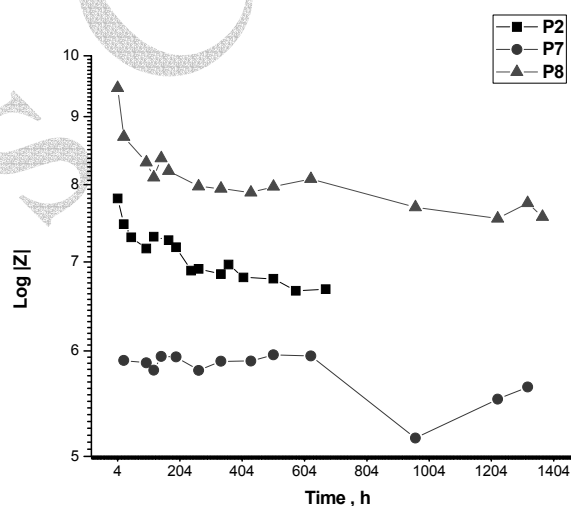


Fig. 7. Development of the impedance module vs. the exposure time for the coatings P-2, P-7, and P-8.

Probably, to the behaviour of this coating contribute not only the higher content of  $ZrO_2$ , but also the higher layer thickness.

The values of the impedance module, and their development with the time, for the coatings P-2, P-7, and P-12 are graphically compared in Figure 7. The behaviour confirms the conclusion established by the polarization measurements, that the referent coating P-2 divides all the investigated coatings into two groups: Monolayer sol-gel coatings with lower and less durable protective ability, and composite coatings, which possess higher and more durable protective ability.

## CONCLUSIONS

New nanocomposite hybrid coatings are developed by sol-gel route as, an alternative to the chromium containing pretreatments, used for protection of the metals against corrosion. The coatings are deposited on basic sol-gel layer with thickness 5–7  $\mu\text{m}$ , over which there is a thin layer of  $\text{SiO}_2$ , (50–100 nm) and two additional layers of EADS. In the bulk of the sol-gel inner layer amorphous  $\text{ZrO}_2$  or crystalline  $\text{CeO}_2$  nanoparticles, are incorporated with particle's sizes of 5 nm and 10 nm, respectively. The AA2024-alloy widely spread in the field of the aviation was used as substrate for all of the samples. The corrosion medium represented 3% NaCl in distilled water.

The protective behaviour of the coatings is evaluated by the use of two independent electrochemical methods independent namely: voltammetry, in its potentiodynamic regime, and Electrochemical Impedance Spectroscopy. By the use of the voltammetric measurements an evaluation of the following parameters is made: the influence of the chemical nature of the inhibitor, its percentage in the sol-gel layer and also the parameters of the process of the coating preparation. Namely, the temperature and duration of the curing process, as well as the layer thickness values. It is demonstrated that the monolayer coatings are inclined to develop pitting corrosion. After the analysis of the impedance spectra in their Bode coordinates, it was established that the monolayer coatings do not supply durable promising protection of the substrate, and consequently, they cannot be used as self-standing coatings. In contrast to them, the multilayer coatings with top-coat of EADS render really a durable and promising protection of the substrate. In their case, no changes in the character of the impedance spectra, even after prolonged exposure are observable.

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## ИЗСЛЕДВАНИЯ ВЪРХУ КОРОЗИОННО-ЗАЩИТНАТА СПОСОБНОСТ НА НАНОКОМПОЗИТНИ ХИБРИДНИ ПОКРИТИЯ

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(Резюме)

Електрохимичното поведение на зол-гелно приготвени наноккомпозитни покрития беше изследвано по отношение на тяхната корозионно-защитна способност. Аморфни  $ZrO_2$  (5 nm) и кристални  $CeO_2$  (10 nm) наночастички бяха внедрени в зол-гелните матрици при съотношение на твърдата компонента от 8% до 20%. Зол-гелните покрития бяха отложени върху подложка от AA 2024. В допълнение, част от тези зол-гелни образци, с цел допълнителна защита, бяха покрити двуслойно с индустриална защитна боя на Европейската агенция по авионавтика и космос (EADS). Дебелините на получените зол-гелни покрития и допълнително защитените образци, съответно, варираха от 13 до 19  $\mu m$  и от 40 до 78  $\mu m$ . Корозионно защитното експлоатационно поведение на тези образци бе определено чрез електрохимични измервания, включвайки волтаперометрия и електрохимична импедансна спектроскопия.