

CHARACTERIZATION OF Ni-P COATING PREPARED ON A WROUGHT AZ61 MAGNESIUM ALLOY VIA ELECTROLESS DEPOSITION

KARAKTERIZACIJA Ni-P PREVLEKE, PRIPRAVLJENE Z NEELEKTRIČNO DEPOZICIJO NA KOVANI MAGNEZIJEVI ZLITINI AZ61

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A low-phosphorous Ni-P coating was prepared on a wrought AZ61 magnesium alloy via electroless deposition for 1 h after an adequate substrate-surface pre-treatment. The prepared coating with a thickness of 10 µm was characterized by the uniform distribution of Ni (95.4 % mass fraction) and P (4.6 % mass fraction) in the cross-section. Microcavities present in the coating resulted in quite a low corrosion resistance of the coated magnesium alloy in a 0.1 M NaCl solution. On the other hand, the coating exhibits a high degree of adhesion, as evidenced by a scratch test, and significantly improves the AZ61 magnesium-alloy microhardness.

Keywords: electroless nickel, magnesium alloy, AZ61, characterization of Ni-P coatings

Malo porozna Ni-P prevleka je bila pripravljena na površini kovane AZ61 magnezijeve zlitine. Prevleka je bila pripravljena z enournu neelektrično depozicijo na predhodno ustrezno obdelani površini zlitine. Pripravljena prevleka debeline 10 µm je imela v prečnem prerezu enakomerno porazdelitev Ni (95,4 % masnih odstotkov) in P (4,6 % masnih odstotkov). Zaradi prisotnosti mikropraznin v izdelani prevleki je korozijska odpornost prevlečene magnezijeve zlitine v 0,1 M NaCl raztopini slaba. Preizkusi razenja prevleke pa so po drugi strani pokazali zelo dober oprijem izdelane prevleke s površino zlitine in znatno izboljšanje mikrotrdote AZ61 magnezijeve zlitine.

Ključne besede: brezelektrično nikljanje, magnezijeva zlitina, AZ61, karakterizacija Ni-P prevlek

1 INTRODUCTION

Due to their low density, magnesium alloys are ranked among the lightest constructional metallic materials. Magnesium alloys concurrently have a high specific strength, toughness and good casting properties. They find their application in the automotive and aerospace industry.¹⁻⁴ A high chemical reactivity, low corrosion resistance and low hardness are their negative properties.⁴ Therefore, it is necessary to protect magnesium alloys against the effects of external environment. There are several ways of protecting magnesium alloys such as galvanic or electroless deposition of coatings, conversion coatings, organic coatings and varnishing.

Electroless-deposited Ni-P coatings improve the coated-material resistance to corrosive environments and material mechanical and wear resistance. Deposited Ni-P coatings have a higher corrosion resistance, physico-mechanical and tribological properties compared to non-treated magnesium alloys.⁵⁻⁷ Generally, the industry identifies three groups of electroless Ni-P coatings according to their phosphorus contents. Low-phosphorus coatings contain 2–5 % mass fractions of phosphorus, medium-phosphorus Ni-P coatings contain 6–9 % mass

fractions of phosphorus and high-phosphorus Ni-P coatings contain 10–13 % mass fractions of phosphorus.⁸ Low-phosphorus Ni-P coatings are predominantly used to increase the hardness of the coated substrate.¹ In general, the hardness, crystallinity and density of electroless Ni-P coatings decrease with the increasing content of phosphorus in the coatings. However, the corrosion resistance of Ni-P coatings increases with the increasing content of phosphorus.^{1,9}

The hardness of deposited low-phosphorus Ni-P coatings can be additionally increased by heat treatment.⁵ During heat treatment, the deposited coating consisting of an amorphous Ni-P phase decomposes due to a gradual increase in the temperature to form crystalline particles of phosphide (Ni₃P). Fine crystalline particles of phosphide (Ni₃P) are formed simultaneously, predominantly in the form of precipitates.⁹ The highest value of the hardness of Ni-P coatings is reached after the heat treatment at 400 °C for 1 h, when the hardness reaches a value of up to 1300 HV.¹⁰ The hardness of Ni-P coatings can also be increased by reducing the phosphorus content by changing the ratio of the components contained in the nickel bath and by changing the coating-process

conditions.¹ However, the Ni-P coating corrosion resistance increases with the increasing content of phosphorus.¹ The phosphorus content in a deposited Ni-P coating can be controlled by adding suitable surfactants into the nickel bath. As mentioned in reference,¹¹ an addition of SDS (sodium dodecyl sulfate) causes an increase in the phosphorus content from 6 % to 9 %. It was observed that the hardness of the Ni-P coating increased with an increase in the SDS concentration up to its critical micelle concentration (CMC) and then decreased. The hardness of the Ni-P coating without a surfactant was 450 HV 0.2 and, in the presence of SDS, the hardness increased up to 685 HV 0.2. The change is due to the change in the coating crystalline structure. In the case of the absence of SDS, the structure of the Ni-P coating is purely crystalline. With an addition of SDS, the structure changes into a mixture of nanocrystalline and amorphous structure. At SDS concentrations higher than CMC, the phosphorus content is 9–10 %. The same effect was observed for CTAB (cetyltrimethylammonium bromide). An addition of CTAB caused an increase of phosphorus in the deposited coating of 7.5–12 % and the hardness of the Ni-P coating increased up to 675 HV 0.2.

In general, the addition of some filler to the Ni-P coating matrix is another possible way to increase the hardness of Ni-P coatings. SiC, Al₂O₃,^{12–13} SiO₂, TiO₂^{14–15} particles and carbon nanotubes can be used as a suitable filler for the Ni-P matrix, improving the coating properties.

The adhesion of the coatings to the substrate is significantly affected by appropriately selected pre-treatments of the substrate surface.¹³ The presented paper deals with the characterization of electroless-deposited Ni-P coatings prepared on an wrought AZ61 magnesium alloy. The influence of the coated-substrate pre-treatment and the coating chemical composition on the coating tribological properties and corrosion resistance was the main objective of the study. A 0.1 M NaCl solution was used for immersion tests to analyze the coated magnesium alloy corrosion resistance and corrosion mechanism.

2 EXPERIMENTAL MATERIAL AND PROCEDURES

2.1 Experimental material

Specimens of the wrought AZ61 magnesium alloy with dimensions of 20 mm × 20 mm × 0.8 mm were used as substrates for the deposition of the Ni-P coating. The EDS-measured chemical composition of the AZ61 magnesium alloy is shown in **Table 1**. The measured chemical composition corresponds to standard ASTM B107M.¹⁶ The microstructure of the alloy is shown in **Figure 1**. The microstructure was documented using a light optical microscope (LM) and the microstructural features were identified using a scanning electron microscope (SEM) with EDS. To reveal the magnesium alloy microstructure, ground and polished metallogra-

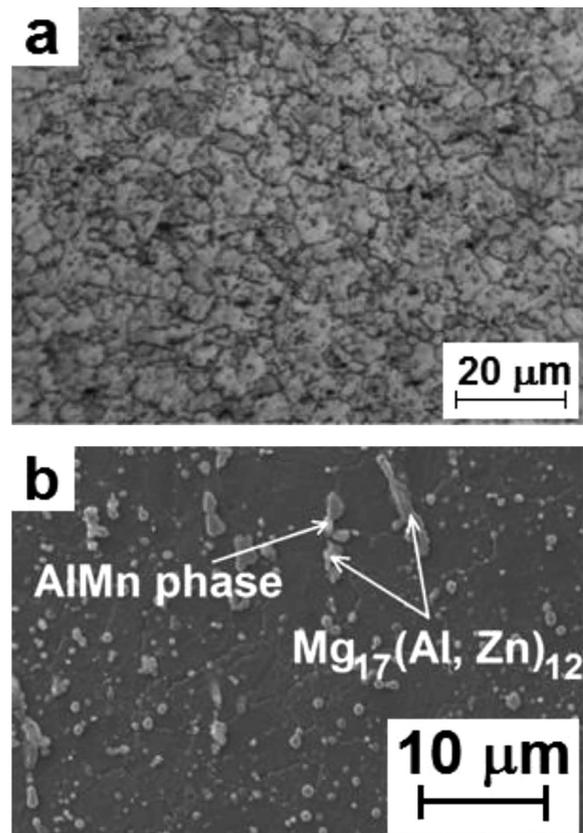


Figure 1: Microstructure of wrought AZ61 magnesium alloy, etched with picral etchant: a) structure of AZ61 magnesium alloy, LM; b) alloy phases, SEM

phic samples were poured into a picral etchant (consisting of 4.2 g picric acid, 10 mL acetic acid, 10 mL water and 70 mL ethanol) for 5 s. The microstructure is formed by δ substitutional solid-solution grains (a solid solution of Al in Mg) and γ particles corresponding to the chemical composition of Mg₁₇(Al,Zn)₁₂. As shown in **Figure 1b**, the presence of AlMn-based intermetallic phases (apparently Al₄Mn) was also evident in the microstructure.

Table 1: Measured elemental composition of the uncoated wrought AZ61 magnesium alloy

Alloy	Elements (w/%)								
	Al	Zn	Mn	Si	Fe	Ni	Cu	Mg	others
AZ61	5.8-7.2	0.4-1.5	0.15-0.5	max. 0.15	max. 0.005	max. 0.005	max. 0.05	bal.	max. 0.3

2.2 Deposition of the Ni-P coating

Before the Ni-P coating deposition, a specific pre-treatment was required to reach the adequate surface roughness and activity. The samples of the AZ61 magnesium alloy were ground using SiC paper no. 1200. Next, the samples were degreased in an alkaline degreasing bath containing soil-releasing agents. The following pickling in an acid pickling bath was performed to activate the surface with partial etching (removing of the

oxide layer). Rinsing the samples with distilled water and isopropanol and drying them with a stream of hot air were performed between the steps of the pre-treatment.

The electroless Ni-P coating deposition, following the sample-surface pre-treatment, proceeded for 60 min. The electroless nickel bath was composed by a nickel source ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$), a reducing agent ($\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$), a complexing agent and an H_2PO_2^- activating substance. The samples were kept in the middle of the bath to ensure a uniform coating formation.

2.3 Characterization of the Ni-P coating

The microhardness of the deposited Ni-P coating was carried out using a Vickers microhardness tester LECO AMH43. Loading of 25 g for 10 s was used for the indentation according to the standard.¹⁷ To obtain the average value, ten indentations were performed on the coating cross-section. The average thickness of the deposited Ni-P coating used for microhardness testing was about 30 μm .

The physicochemical properties of the deposited Ni-P coating were evaluated using a CSM Instruments REVETEST scratch tester with the progressive-load-type method and a Rockwell diamond indenter with the top angle of 120° and the top radius of 200 μm . For the evaluation of the physicochemical properties, the surface of the substrate was polished to the roughness of $R_a \approx 0.25 \mu\text{m}$ using diamond pastes during the pre-treatment after the grinding. The friction force, the friction coefficient, the penetration depth and the acoustic emission were recorded along with the adhesion during the scratch test. The applied normal force was set in a range of 1–20 N. The speed of the indenter was 1.58 mm min^{-1} and the total length of the trace was 3 mm.¹⁸

A Zeiss Evo LS-10 scanning electron microscope (SEM) equipped with an EDS Oxford Instruments Xmax 80 mm² detector and the AZtec software was used to determine the average contents of nickel and phosphorus in the deposited Ni-P coatings. SEM observations were also used to evaluate the mechanism of the corrosion degradation of the magnesium substrate and the deposited Ni-P coating after an exposure to 0.1 M NaCl.

For the evaluation of the mechanism of the corrosion degradation of the magnesium substrate and the deposited Ni-P coating, the samples were immersed into a 0.1 M solution of NaCl for 20 min. After this time, the surface of a sample was analyzed using the scanning electron microscope and the mechanism of corrosion degradation was determined on the cross-section of the sample.

3 RESULTS AND DISCUSSION

3.1 Characterization of the deposited Ni-P coatings

Uniform Ni-P coatings were deposited on the AZ61 magnesium alloy substrate. **Figure 2a** shows the nodular

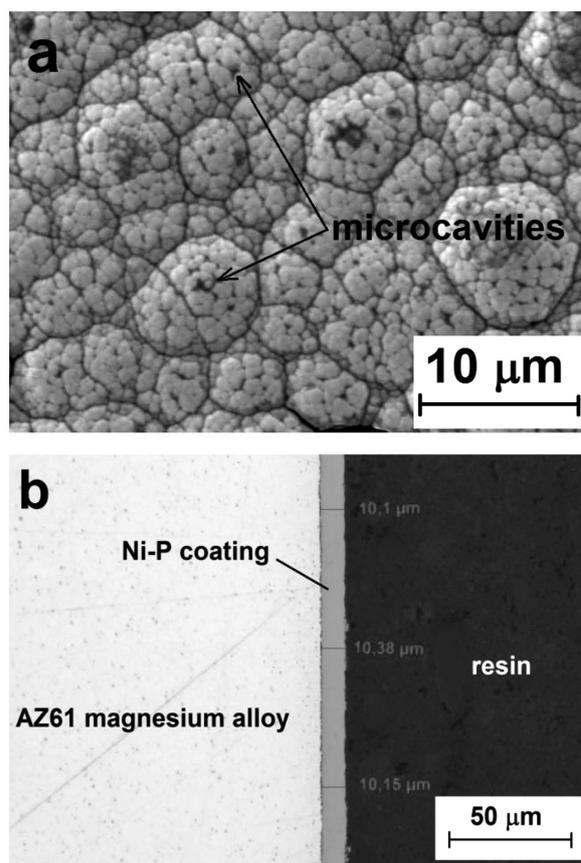


Figure 2: Microstructure of Ni-P coating: a) surface morphology of Ni-P coating, b) cross-section morphology of Ni-P coating on AZ61 magnesium alloy

structure of the Ni-P coating with a typical cauliflower-like pattern. Between these nodular cusps, a certain amount of microcavities is present. These microcavities are nucleation sites for micropitting in the case of material exposure to a corrosive environment. However, no macrodefects were observed in the deposited Ni-P coatings, neither at the Ni-P/substrate interface. **Figure 2b** shows the cross-section morphology of the deposited Ni-P coating. It shows that the Ni-P coating is uniform and compact. The average thickness of the deposited Ni-P coating used for the microhardness testing was about 30 μm and the average thickness of the deposited Ni-P coating used for the EDS analysis and the scratch test was about 10 μm .

Using the EDS analysis, it was determined that the content and distribution of individual components in the deposited Ni-P coating were homogeneous throughout the entire cross-section (**Figure 3**). The analysis showed that the nickel content in the deposited Ni-P coating was 95.4 ± 0.1 % mass fraction and the phosphorus content was 4.6 ± 0.1 % mass fraction. Based on references,^{8,10} this coating can be classified as a low-phosphorus Ni-P coating. The homogenous distribution of individual elements in the coating is a sign of a continual coating growth.

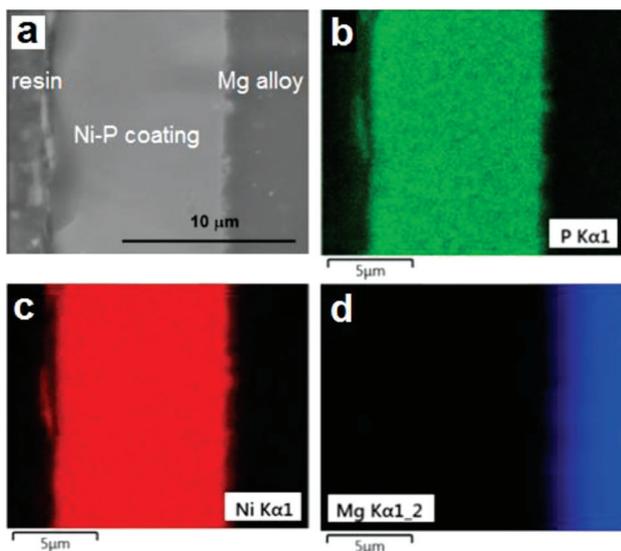


Figure 3: Mapping of Ni-P coating on AZ6 alloy: a) structure of Ni-P coating, b) phosphorus, c) nickel, d) magnesium

3.2 Mechanical and physicochemical properties of the deposited Ni-P coating

The resulting average microhardness value of the prepared electroless-deposited low-phosphorous Ni-P coating is 700 ± 40 HV 0.025, as measured on 10 random places on the coating cross-section. The microhardness of the plain magnesium substrate is 79 ± 6 HV 0.025, so the microhardness of the Ni-P coating is around nine times higher than that of the AZ61 magnesium substrate.

The obtained value is higher when compared to the microhardness of the coating reported on in reference¹⁹. On the other hand, a positive influence of the filler addition and heat treatment on the coating microhardness was observed in reference¹⁹. An increase in the microhardness from 380 ± 10 HV 0.1 to 540 ± 10 HV 0.1 was observed when adding SiO₂ nanoparticles to the Ni-P coating¹⁹; however, this value is still lower when compared to the presented coating. The microhardness of the electroless-deposited composite coating reported on in reference¹⁹ increased to 970 ± 10 HV 0.1 when applying a heat treatment to the coated component at 400 °C.

A similar effect was observed in the work reported in reference²⁰ where the hardness of the plain Ni-P coating reached a value of 608 ± 12 HV. With the addition of a TiO₂ colloidal solution to the nickel bath, followed by a co-deposition of TiO₂ particles into the Ni-P coating, the hardness value of the Ni-P/TiO₂ composite coating increased to 685 ± 18 HV, which is comparable with the presented coating. The increase in the hardness of the deposited Ni-P/TiO₂ composite coating to 1325 ± 40 HV was observed by following the application of a heat-treatment.²⁰

The obtained values of critical normal forces L_{c1} and L_{c2} and adequate values of friction forces F_{t1} and F_{t2} at normal forces L_{c1} and L_{c2} , respectively, are shown in

Table 2. The record of the scratch test of the Ni-P coating on the AZ61 magnesium alloy is shown in **Figure 4**. Details of the Ni-P coating on the AZ61 magnesium alloy after the scratch test for normal forces L_{c1} and L_{c2} are shown in **Figures 5a** and **5b**, respectively.

The determined value of critical normal force L_{c1} was 6.9 N. The formation of oblique and parallel cracks were observed at L_{c1} , **Figure 5a**. The determined value of critical normal force L_{c2} was 11.9 N. The formation of transverse arch cracks was observed at L_{c2} , **Figure 5b**. Ductile failure of the coating due to the internal tensile stresses occurs during the scratch test.

Table 2: Values of critical normal forces and friction forces of Ni-P coatings and a comparison of these values with the published data

Lit.	Substrate	Coating	L_{c1} (N)	L_{c2} (N)	F_{t1} at L_{c1} (N)	F_{t2} at L_{c2} (N)
This work	AZ61	Ni-P	6.9	11.9	0.8	2.2
(21)	AZ31	Ni-P	7.3	12.3	1.1	2.6
(2)	AZ91	Ni-P	-	17.6 (L_c)	-	-
(23)	AZ61	PEO	2.69 ± 0.10	-	-	-
(24)	AZ61	Ti/Ti (C,N)/ (TiAl)N	3	10	-	-

Comparing critical loads L_{c1} and L_{c2} of the experimental Ni-P coating deposited on the AZ61 magnesium alloy with the Ni-P coating deposited on the AZ31 magnesium alloy from²¹, it became clear that the Ni-P coating on the AZ61 magnesium alloy achieved lower critical values L_{c1} and L_{c2} . Oblique, parallel and transverse arch cracks were also observed on the Ni-P coating on the AZ31 magnesium alloy.²¹ This effect can be attributed to a slight difference between the methods of the pre-treatment of the magnesium substrate before the deposition process.

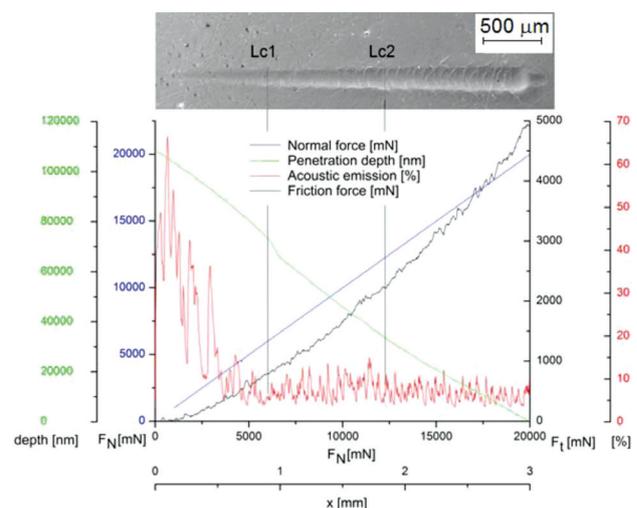


Figure 4: Evaluation of scratch tests for Ni-P coating on AZ61 magnesium alloy

The adhesion of the deposited heat-treated Ni-P coating prepared on the AZ91 magnesium alloy was evaluated in reference.² The first cracks on the heat-treated Ni-P coating deposited on the AZ91 magnesium alloy were observed at a load of 17.6 N and their characteristics were the same for all the heat-treated samples. However, the character of the cracks is slightly different compared to the experimental samples (Ni-P on AZ61) due to the heat treatment of the Ni-P coating. Heat-treated coatings are more brittle compared to the non-treated coatings.¹ Moreover, it was observed that the abundance of cracks increased with the increasing applied load.

In literature², critical load value L_c , where the first cracks were observed, is higher than that of the Ni-P coatings deposited on the AZ61 magnesium alloy (**Table 2**) and the samples of AZ31 described in reference²¹. This fact can be attributed to several factors. The progressive-load-type method of the scratch test was chosen for the evaluation of the adhesion of the experimental Ni-P coating on an AZ61 alloy and the Ni-P coating on an AZ31 alloy in ²¹. However, the constant-load-type method of the scratch test was chosen for the evaluation of the adhesion of a heat-treated Ni-P coating in literature ². Moreover, the initial load for the evaluation of the heat-treated Ni-P coatings on the AZ91 magnesium alloy was determined to be 8.80 N and the load was increased five times to 44.0 N. The heat treatment can affect the

adhesion of the coating to the substrate. As indicated in literature²², the creation of Al-Ni intermetallic phases can significantly reduce the adhesion of Ni-P coatings to the AZ91 magnesium alloy after the heat treatment. However, this effect was not observed in the research from reference². This can be attributed to the fact that the presence of these phases is limited to small areas of the γ phase ($Mg_{17}Al_{12}$) present on a substrate surface. The content of the γ phase is dependent on the Al content in the substrate. Clearly, a low amount of Al in AZ91 used in the research from reference² did not have a detrimental effect on the coating adhesion.

As shown in **Table 2**, it was observed that the adhesion of the PEO (plasma electrolytic oxidation) coating on the AZ61 magnesium alloy mentioned in reference²³ is lower with respect to the Ni-P coatings deposited on the AZ61 magnesium alloy in this work. In particular, a significantly lower value of critical load L_{c1} resulting in coating damage was measured in the research from literature²³, with respect to the present work.

It can be noted that the deposited Ni-P coating showed a higher adhesion to the magnesium-alloy substrate with respect to the PEO or plasmatic composite coatings.²⁴ These composite coatings and plasma-deposited layers are more brittle than the deposited coatings. In addition, the adhesion of coatings to the substrate is negatively affected by the stresses at the matrix/filler interface created during the coating formation.²⁵

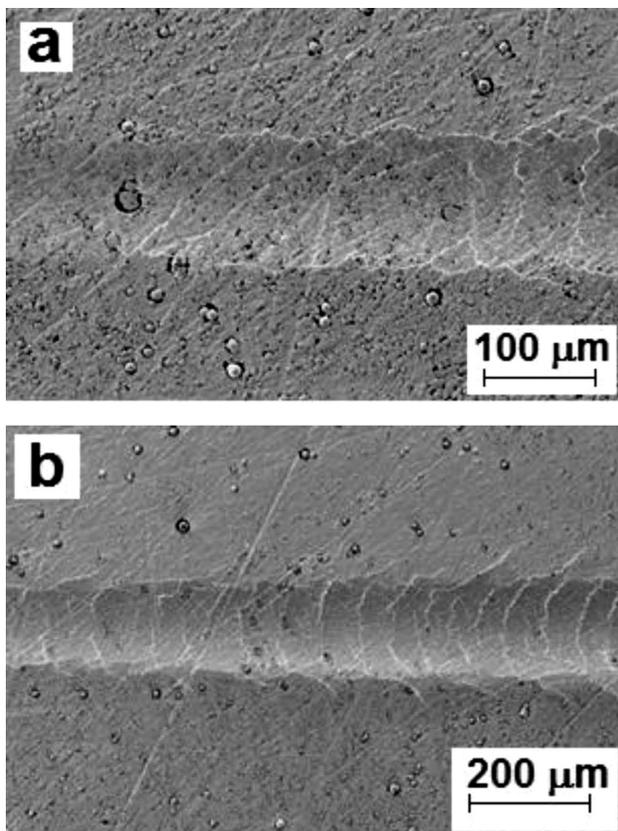


Figure 5: Details of Ni-P coating after scratch tests: a) L_{c1} , b) L_{c2}

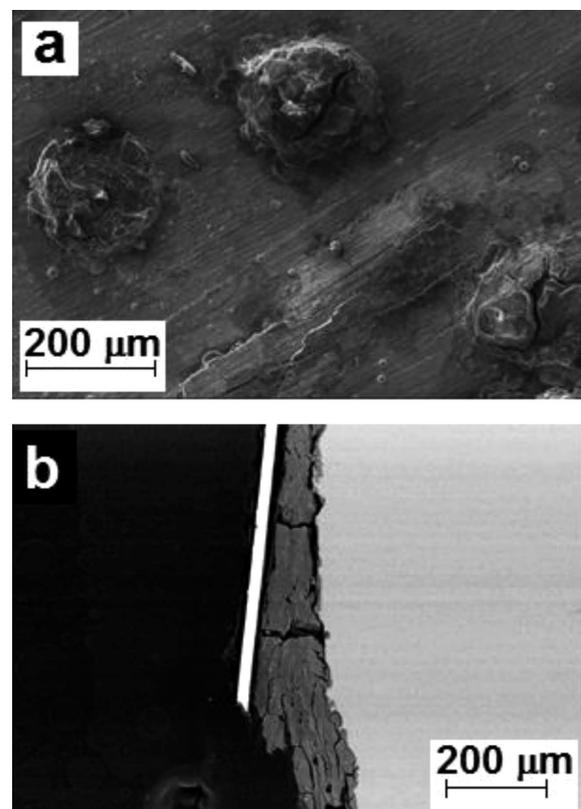


Figure 6: Ni-P coating on AZ61 magnesium alloy after an exposure to 0.1 M NaCl: a) surface, b) cross-section

3.3 Exposure of the samples to a corrosive environment

Figure 6 shows a corrosion degradation of the AZ61 magnesium alloy with the Ni-P coating after an exposure to a 0.1 M NaCl solution for 20 min. The low-phosphorus coatings are characterized by a higher hardness and crystallinity but also by a lower corrosion resistance when compared to the high-phosphorous Ni-P coatings.¹⁰

The deposited Ni-P coating was characterized by a structure with nodular cusps (**Figure 2a**). An imperfect nickel deposition may occur among these cusps, causing the Ni-P coating to have a certain amount of microcavities in its volume. These microcavities are the nucleation sites for a corrosion attack and micropitting in the case of an exposure to a corrosive environment. The transport of corrosive agents to the surface of the magnesium substrate occurs through these microcavities. The corrosion process starts at the magnesium substrate/Ni-P coating interface. Chemical reactions occur due to the interaction between the corrosive agents and the magnesium-alloy substrate. The formed corrosion products accumulating on the magnesium alloy under the Ni-P coating lead to a local destruction of the coating as shown in **Figure 6**. An EDS analysis revealed that these corrosion products are predominantly formed by the oxides and chlorides of magnesium.

The presence of microcavities in Ni-P coatings can be eliminated or removed by adding suitable surfactants to the nickel bath. Such suitable surfactants can be sodium dodecyl sulfate, sodium benzenesulfonate or CTAB.^{11,26–27}

4 CONCLUSION

An electroless-deposited low-phosphorous Ni-P coating was successfully prepared on a wrought AZ61 magnesium alloy by applying a pre-treatment process. The deposited Ni-P coating was characterized in terms of elemental composition, mechanical and physicochemical properties.

In terms of the chemical composition, the deposited 10- μm -thick Ni-P coating treated for 1 h shows a high degree of homogeneity over the entire cross-section with a phosphorus content of $4.6\pm 0.1\%$ and a nickel content of $95.4\pm 0.1\%$. The microhardness of the deposited Ni-P coating reached a value of $700\pm 40\text{ HV } 0.025$. In terms of physicochemical properties, the deposited Ni-P coating was characterized by a high degree of adhesion. The adhesion was determined from the corresponding critical normal forces L_{c1} (6.9 N) and L_{c2} (11.9 N). The mechanism of corrosion degradation of Ni-P applied on the AZ61 magnesium alloy substrate was determined based on a metallographic observation of a corrosion attack with the subsequent degradation of the magnesium substrate due to the microcavities present in the coating, allowing the contact of the corrosive environment with the substrate.

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