AN ACTIVATION OF ELECTROLESS PROCESS OF Ni-P DEPOSITING ON AUSTENITIC STAINLESS STEEL

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Abstract

Since, it is very difficult to form Ni-P deposit by electroless process on austenitic stainless steel as substrate, the activation with a weak acid etch, i.e., nickel strike should be applied. In this work a new method for Ni-P depositing on austenitic stainless by electroless processing is developed. In this way was avoided nickel strike pre-coating treatment which makes Ni-P coating process on stainless steel more complicated in comparison to other similar electroless processes on other types of steel, aluminium alloys, and so on. Study has been focused on influence of new activation process on adhesivity, microhardness and microstructure analysis of electroless Ni-P coatings. Adhesivity was estimated by Vickers indenter. Microhardness was tested by using scanning electron microscope. Microstructure of the electroless treated specimens were analyzed by optical and scanning electron microscopy. Based on experimental results, it can be concluded that by new method of activation of surface, electroless nickel - phosphorous coatings on austenitic stainless steel have higher adhesivity and microhardness than non-activated electroless nickel - phosphorous coatings.

Keywords: Electroless, coatings, stainless steel, micro-hardness, adhesivity

1. INTRODUCTION

Generally, an electroless Ni-P layer has a higher hardness and a better corrosion resistance than the AISI 316 stainless steel [1]. But, it is very difficult to form a Ni-P deposit on an austenitic stainless-steel as substrate using the electroless process. For that reason, the activation of surface with a weak acid etch, i.e., nickel strike should be applied [2]. Nickel-strike pre-coating treatment makes the Ni-P coating deposition on stainless steel more complicated in comparison to the other similar electroless depositions on other types of steel, aluminium alloys and so on. The heat treatment should be applied after the electroless coating process. Ni-P alloy layers should be heat treated, mainly to increase hardness and adhesion of Ni-P alloy layers with substrate [2].

Microstructure of the Ni-P coatings deposited with the electroless process depends on the phosphorous content. Electroless deposited Ni-P layers are crystalline if the phosphorus content is between 1-5 % mass fractions (low phosphorus). If the content of phosphorous is between 6-9 % mass fraction (medium phosphorous), the Ni-P layers deposited with the electroless process have mixed, amorphous and crystalline structures. If the content of phosphorous is between 10-13 % mass fraction (high phosphorus), the Ni-P layers deposited with the electroless process are amorphous [3,4-8].

With a prolonged aging at high temperatures, amorphous electroless deposited nickel-phosphorous layers begin to crystallize and lose their preferable amorphous character. At the same time, a higher hardness of the layer is obtained due to the diffusion of phosphorus from the region near the interface with the substrate [9]. The nickel-phosphide particles conglomerate and the matrix of Ni_3P forms due to the continued heating [9] The hardness of the layer can increase with the appearance of the intermetallic Ni_3P phase and with a higher crystallinity of the nickel-phosphorous layers [2,4,5,10]. Moreover, the hardness of the electroless deposited nickel-phosphorous layers can increase because of the precipitation of the Ni_3P phase [10]. The maximum hardness can be obtained if the phosphorus content is around 4 % mass fraction [3,4-8].

The microhardness of electroless Ni-P layers depends on the heat treatment, the content of phosphorus and on the contents of other alloying elements in the coatings [3,4,11-14]. After the heat treatment, the structure of the coating is more crystalline; moreover, the intermetallic nickel phosphide (Ni_3P) phase appears [4,5,10]. Nickel with an amorphous structure has a lower hardness than nickel with a crystalline structure [5-7,13,15-27]. The grain size of electroless Ni-P composite layers can have a significant influence on the hardness [19]. Heat treated of Ni-P layers deposited with the electroless process can have a significant impact on their hardness and adhesivity [8]. In this work study has been focused on influence of new activation process on adhesivity, microhardness and microstructure analysis of electroless Ni-P coatings. The adhesivity related to the optimization of electroless Ni-P process was estimated with a Vickers indenter and Rockwell indenter.

2. AN OPTIMIZATION OF ACTIVATION OF ELECTROLESS PROCESS OF NI-P DEPOSITING ON AUSTENITIC STAINLESS STEEL

The activation electroless process was done by immersing a special aluminium electrode in the electrolyte together with treated cylindrical specimen of 8 mm in diameter 50 mm in length. The chemical composition of austenitic steel AISI 316 is shown in **Table 1**.

Table 1 Chemical composition of steel substrate

Chemical composition in mass fraction (*w*/%)							
C	Si	Mn	P	S	Cr	Mo	Ni
0.07	0.71	1.36	0.031	0.021	17.1	2.42	11.6

The Nikora nickel bath based on an aqueous solution of sodium hypophosphite (a registered trademark of Schering AG, Berlin) was used. The surfaces of specimens were cleaned to eliminate all types of surface contamination before the electroless process. Specimens were mechanically polished using Kemipol T-12, with Al_2O_3 grains of 14 µm. After that the degreasing the surfaces of the samples with the cleaning agent UNICLEAN 253, which is composed of silicate, hydroxide and biodegradable surfactants. The substrate surfaces were washed and activated in the activation agent UNICLEAN 675. After rinsing, the main electroless-deposition process was applied (**Figure 1**).

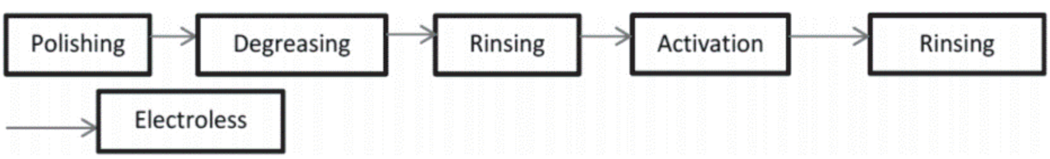

Figure 1 Flow-chart diagram of the electroless process of nickel plating on an austenitic-steel AISI 316 substrate

After the electroless processing, samples were heat treated by aging at 500 °C for 60 min in an air-furnace atmosphere [17].

An analysis of the Ni-P coating layers was carried scanning electron microscope FEG FEI QUANTA 250 SEM. The contents of the iron, chromium, nickel and phosphorus of the non-heat-treated sample were evaluated with SEM and EDS mapping. A map of the contents of iron, chromium, nickel and phosphorus is shown in **Figure 2**.

It is evident that nickel (**Figure 2c**) and phosphorous (**Figure 2d**) are located in both the coating and the substrate. Iron (**Figure 2a**) and chromium (**Figure 2b**) are located only in the substrate. Phosphorous is uniformly distributed in the Ni-P coating.

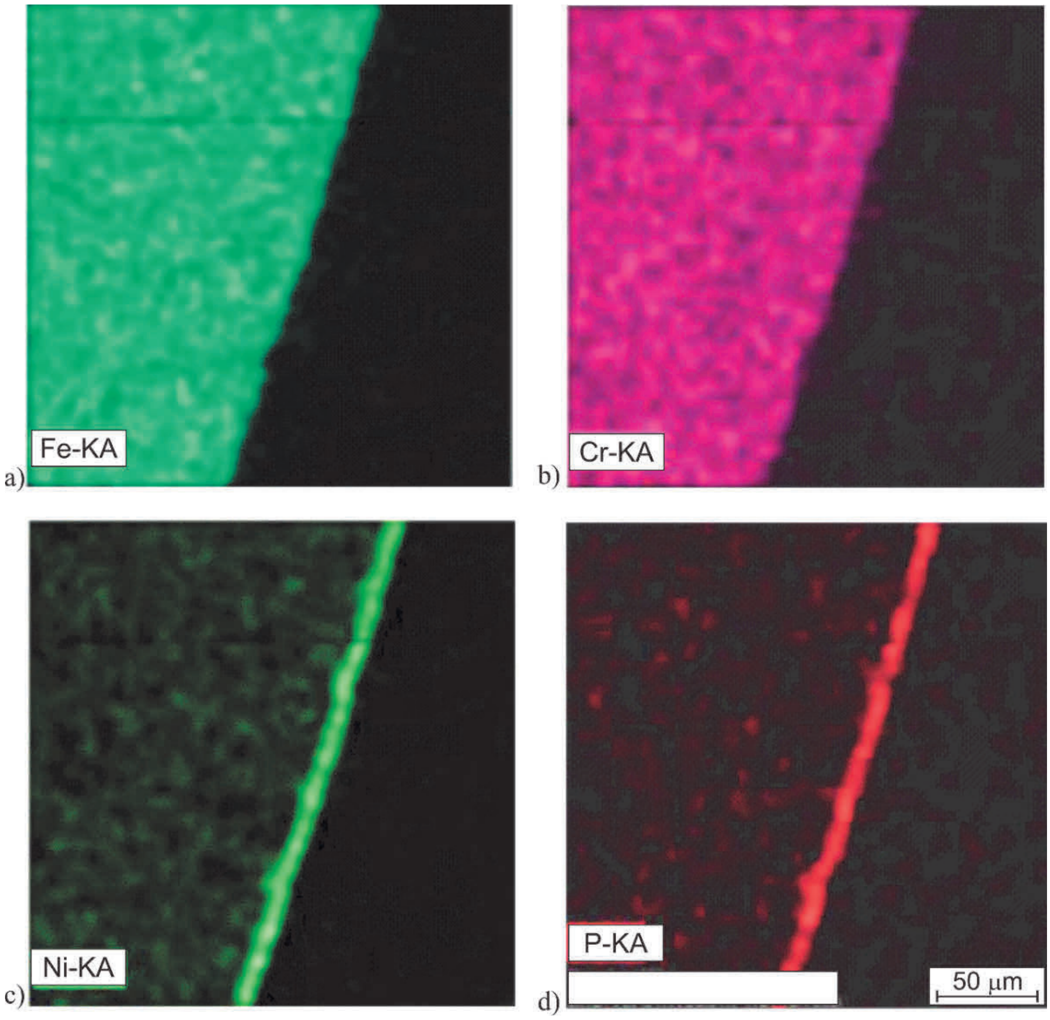

Figure 2 SEM and EDS mapping of the iron, chromium, nickel and phosphorus of the non-heat-treated samples

SEM and EDS mapping of the contents of the iron, chromium, nickel and phosphorus in the heat-treated samples is shown in **Figure 3**. It is evident that the distribution of chemical elements in the coating and the substrate is similar to the distribution of chemical elements in the non-heat-treated specimen. Nickel (**Figure 3c**) and phosphorous (**Figure 3d**) are found in the coating and in the substrate, but iron (**Figure 3a**) and chromium (**Figure 3b**) are found only in the substrate. It was found that the heat-treated and non-heat treated specimens have about 9 % of phosphorous. The deposited layer could be recognized by his light color, opposite to the dark colored substrate. The thickness of the non-heat-treated Ni-P coating is 8 µm while the thickness of the heat-treated Ni-P coating is 7.5 µm.

Samples were tested with the microhardness indentation technique. The Vickers microhardness of each sample was obtained by the Vickers tester Struers Duramin. The obtained microhardness of the non-heat-treated electroless Ni-P coating on the austenitic stainless-steel AISI 316 substrate was 429 ±17 HV0.01, while the hardness of the heat-treated electroless Ni-P coating was 853 ±26 HV0.01.

The adhesivity was estimated with a Vickers indenter.

It can be seen in **Figure 4** that the delamination of the deposited layer did not appear on the specimen treated with special pre-coating activation of specimen surface. The application of heat treatment without special activation of surface before electroless processing of specimens is not enough for satisfied adhesion of electroless deposited layer with substrate.

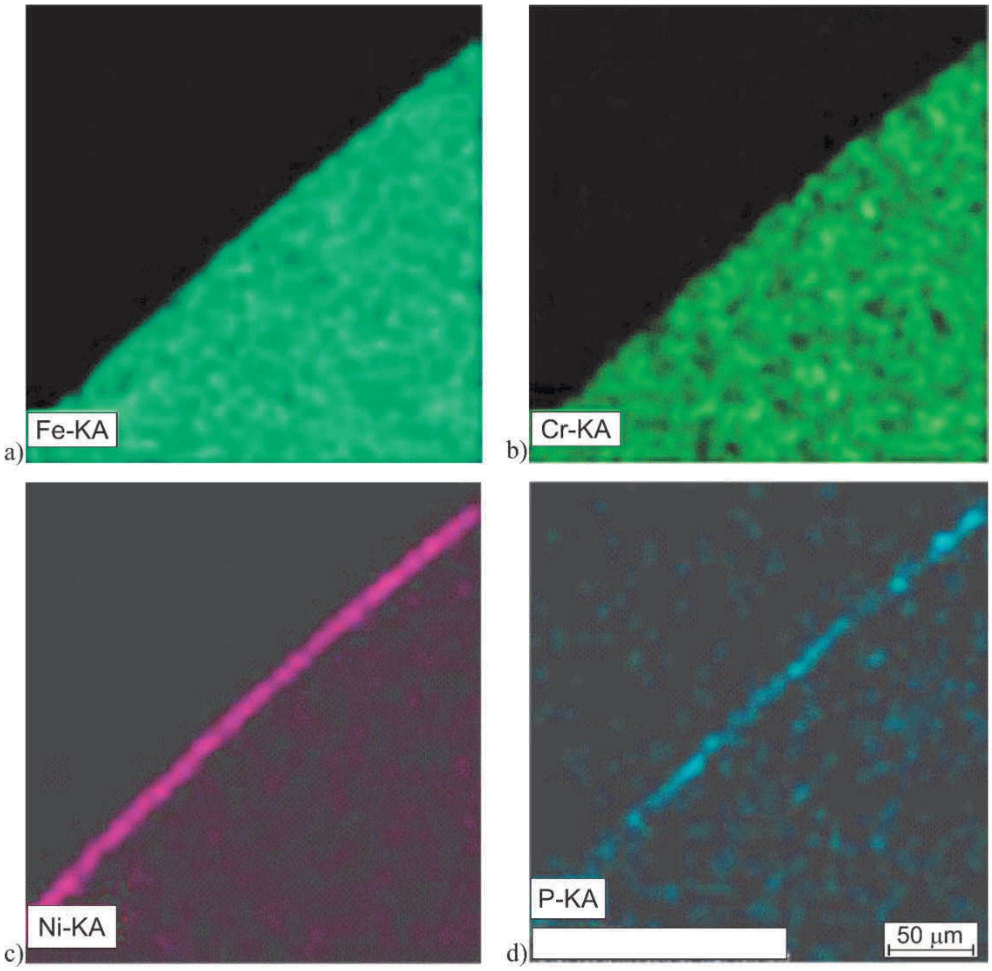

Figure 3 Content of the iron, chromium, nickel and phosphorus present on the cross-sections of Ni-P coatings deposited with the electroless process on austenitic stainless-steel AISI 316 substrate of the heat-treated sample, obtained with SEM and EDS mapping

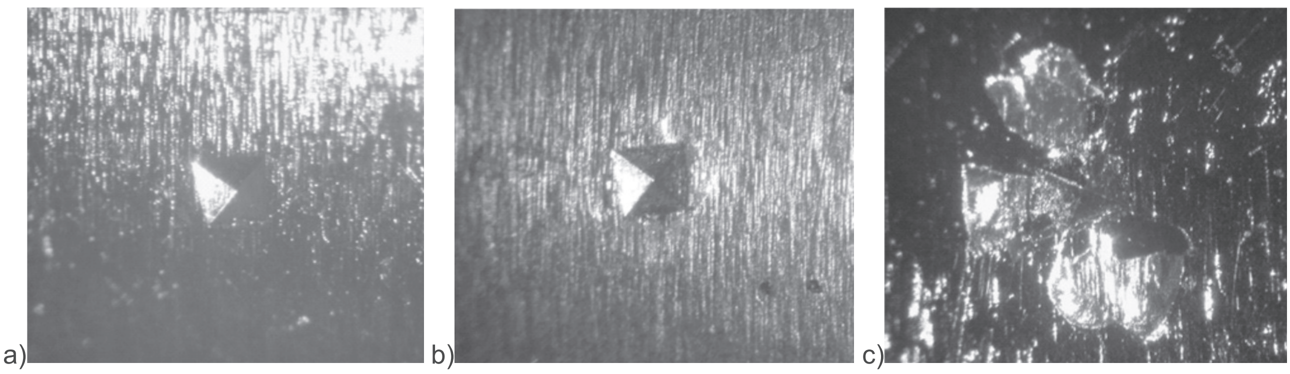

Figure 4 Indentation results for adhesivity of Ni-P electroless coatings, mag. 35:1: a) chemical pre-coating treatment of the surface + electroless coating, b) pre-coating treatment of the surface + electroless coating + aging at 500 °C, c) electroless coating + aging at 500 °C

3. CONCLUSION

Application of the Ni-P coatings deposited with the electroless process on the austenitic steel AISI 316 was analysed. Surfaces of the austenitic-steel AISI 316 substrate were prepared before depositing the Ni-P coatings with the electroless process. The investigated coatings follow the surface morphology of the samples. Ni-P coatings deposited with the electroless process were formed chemically uniform. The thickness of electroless deposited is not depending on application of heat treatment.

By the Vickers microhardness testing it was found out that bay application of heat treatment can to achieve better results. The applicability of Vickers indenter in testing and optimization of adhesivity of electroless Ni-P coatings deposited on austenitic stainless steel AISI 316 was analyzed. Based on Vickers indentation results of adhesivity of Ni-P electroless coatings it can be concluded that by application of proper activation process before electroless coating a satisfied adhesivity can be achieved.

ACKNOWLEDGEMENTS

This work has been supported in part by Croatian Science Foundation under the project 5371.
This work has been supported in part by University of Rijeka, Support No 13.09.1.1.02.

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