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Adhesivity of electroless Ni-P layer on austenitic stainless steel

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ABSTRACT

Purpose: Optimization of testing of adhesivity of electroless deposited nickel-phosphorous coatings on austenitic stainless steel substrate.

Design/methodology/approach: The main study has been focused on comparison of testing methods of adhesivity of electroless nickel-phosphorous coatings and analyses of influence of heat treatment on adhesivity of electroless nickel-phosphorous.

Findings: It was found out that adhesivity of electroless nickel-phosphorous coatings can be successfully characterized by Vickers hardness tester.

Research limitations/implications: The study was not extended to other types of deposited layers in order to be able to bring a general decision about applicability of this method in the testing of adhesivity of deposited layers.

Practical implications: Based on experimental results it was found out that heat treated nickel-phosphorous coating have higher microhardness than non-heat treated electroless nickel-phosphorous coatings. High microhardness of heat treated nickel-phosphorous coating is connected with formation of Ni_3P phase. Moreover, better adhesivity can be achieved by application of proper activation process before electroless coating.

Originality/value: Standard Vickers hardness method was successfully applied in testing of adhesivity of electroless nickel-phosphorous coatings on austenitic stainless steel substrate.

Keywords: Electroless; Deposition; Coatings; Heat treatment; Stainless steel; Adhesivity

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PROPERTIES

1. Introduction

Electroless deposited coatings have a more uniform thickness on a complex-shaped objects in comparison to electroplated deposits. This uniform thickness and composition of the coating results in uniform mechanical and physical properties of surface layer [1, 2, 3]. Generally, electroless Ni-P coating has higher hardness and better corrosion resistance than AISI 316 stainless steel [4].

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 [5]. Nickel strike pre-coating treatment makes Ni-P coating process on stainless steel more complicated than Ni-P coating processes on other types of steel, aluminium alloys, and so on. The heat treatment should be applied after electroless coating process [5]. Ni-P alloy coatings should be heat treated preferably to increase hardness of Ni-P alloy coatings, but recent investigations have showed that adhesivity of electroless Ni-P coating can improved by heat treatment application.

Generally, microstructure of the Ni-P coatings deposited by electroless process depends on the phosphorous content. Electroless deposited Ni-P coatings will be crystalline if the phosphorus content is between 1 to 5 wt.% (low phosphorus). If the content of phosphorous is between 6 to 9 wt.% (medium phosphorous), Ni-P coatings deposited by electroless process consist of mixed amorphous and crystalline structures. If the content of phosphorous is between 10 to 13 wt.% (high phosphorus), Ni-P coatings deposited by electroless process are amorphous [1,6-9].

To achieve high adhesion, thorough surface preparation, or removing foreign contaminants from the base metal surface and eliminating mechanically distorted surface layers to present a clean, healthy surface structure is required [10]. With prolonged heat treatment, i.e., aging at high temperatures, electroless deposited nickel-phosphorous coatings begin to crystallize and lose its preferable amorphous character [11]. At the same time higher hardness on stainless steel is obtained. As suggested by the authors in ref. [11], this effect exists probably due to diffusion of the phosphorus from the region near the interface into the substrate. With prolonged heat treatment at high temperatures the nickel phosphide particles conglomerate and the matrix of Ni_3P forms with continued heating [11]. Hardness of coating can increase by appearance of intermetallic Ni_3P phase, and by higher crystallinity of nickel-phosphorous coatings [5-7,12]. Moreover, hardness of electroless deposited nickel-phosphorous coatings can increase because of the precipitation of Ni_3P phase [12]. The maximum hardness can be obtained if the phosphorus content is around 4 wt.% [1,6-9].

Since adhesivity of electroless Ni-P coating is not so high the standard VDI 3198 sometimes is not sensitive enough in testing of Ni-P coatings [13]. In this work, adhesivity of electroless Ni-P coating was tested was performed by Vickers indenter.

2. Experimental work

In applied experimental procedure work, cylindrical specimens of austenitic steel AISI 316 were used as substrate. Chemical composition of steel specimens is shown in Table 1.

Table 1.
Chemical composition of steel substrate

Chemical composition/wt.%							
C	Si	Mn	P	S	Cr	Mo	Ni
0.07	0.71	1.36	0.031	0.021	17.1	2.42	11.6

Diameter of cylindrical specimens was equal to 8 mm and their length was equal to 50 mm. Before electroless process, surface of specimens has been cleaned to eliminate all types of surface contaminations. At first, specimens were mechanically polished by using Kemipol T-12, with Al_2O_3 grains of 14 μm. This has been followed by degreasing the surface of the sample by cleaning agent UNICLEAN 253, which is composed of the silicate, hydroxide and biodegradable surfactants. After that, the substrate surfaces were washed and activated in activation agent UNICLEAN 675. Additional activation was done by chemical pre-coating treatment. After rinsing, the main electroless deposition process has been applied (Figure 1).

Electroless nickel plating process has been carried out by nickel bath Nikora (registered trademark of Schering AG, Berlin). It is known that the nickel bath Nikora is based on an aqueous solution of sodium hypophosphite. Chemical composition of the electroless plating bath has not been studied.

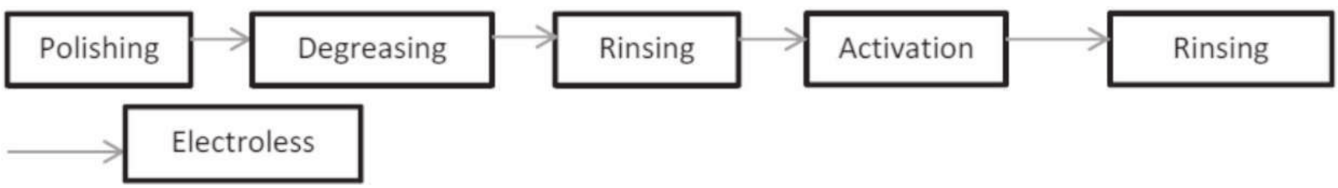

Fig. 1. Flow chart diagram of the electroless process nickel plating on austenitic steel AISI 316 substrate

After the electroless processing, one part of samples has been heat treated by aging at 500 °C for 60 min in air furnace atmosphere [14]. Other part of samples has not been heat treated after the electroless processing. Heat treatment was conducted not longer than 24 hours after applying the electroless processing on specimens.

Ni-P coating layers of non-heat treated substrate and heat treated samples have been tested by microhardness indentation technique. The Vickers microhardness testing of each sample has been determined as the average of five test results by Vickers tester Struers Duramin 2. Microstructure analysis of Ni-P coating layers were obtained by scanning electron microscope FEG FEI QUANTA 250 SEM. X-ray Diffraction (XRD) analysis of the heat treated electroless coating was carried out by instrument BRUKER AXS D8-Advance, Vertical Theta-Theta goniometer with Co radiation

3. Results and discussion

The obtained microhardness of the non-heat treated electroless Ni-P coating on 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.

No relevant differences between thicknesses of those two Ni-P coatings has been detected. The thickness of the non-heat treated Ni-P coating is equal to 8.11 ± 0.18 μm while the thickness of the heat treated Ni-P coating is equal to 7.52 ± 0.18 μm (Figure 2).

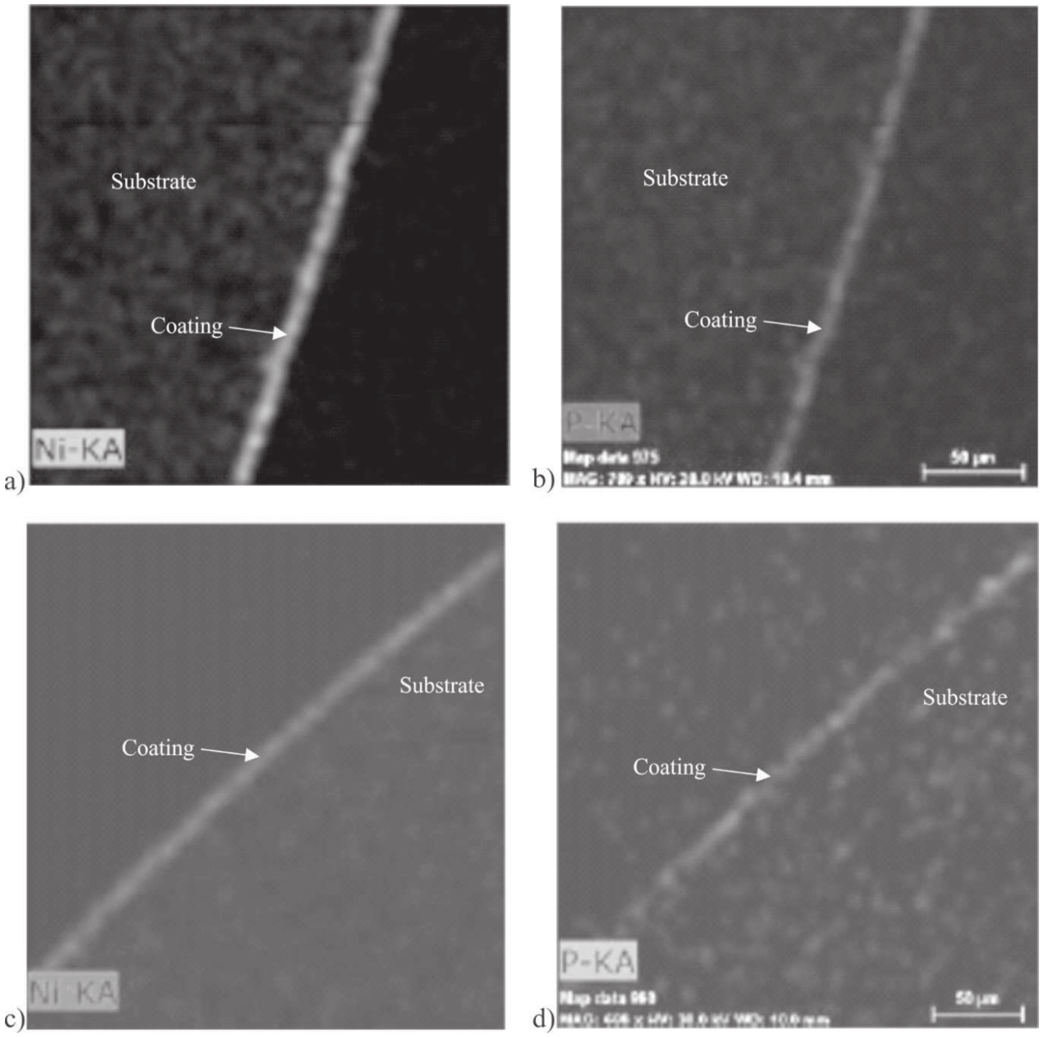

Fig. 2. SEM and EDS mapping of the a) nickel and b) phosphorus of the non-heat treated sample, and c) nickel and d) phosphorus of the heat treated sample on the cross section of Ni-P coatings deposited by electroless process on austenitic stainless steel AISI 316 substrate

SEM and EDS mapping of the nickel and phosphorus on the cross section of Ni-P coatings deposited by electroless process on austenitic stainless steel AISI 316 substrate is shown in Figure 2. No relevant differences between distribution of chemical composition in the heat treated and non-heat treated electroless Ni-P coatings has been detected.

Content of the nickel and phosphorus of the non-heat treated samples are shown in Figure 2a and Figure 2b, respectively, and content of the nickel and phosphorus of the heat treated samples are shown in Figure 2c and Figure 2d, respectively. It is evident that nickel (Figure 2a and 2c) and phosphorous (Figure 2b and 2d) are located in both, coating and substrate.

It was obtained that heat treated and non-heat treated specimens have about 9 % of phosphorous. Distributions of nickel and phosphorous in coating are similar in both, heat treated and non-heat treated specimens, but it is known that non-heat treated coating with 9 % of phosphorous has mixture of amorphous and crystalline structure [5].

For the phase identification laboratory X-ray diffraction analysis was performed on the specimen's surface after applying electroless procedure by utilizing a Philips X'Pert Multi-Purpose Diffractometer (PANalytical) in Bragg-Brentano geometry. While obtaining the X-ray diffraction pattern the specimen was rotated around the surface normal in order to acquire better crystallite statistics. The presence of Ni_3P phase was confirmed on the basis of the reflections which are expected in view of its tetragonal crystal structure.

Indeed, the X-ray diffraction pattern (Figure 3) clearly shows presence of the Ni_3P phase on the surface of the specimen which was subjected to the heat treatment procedure.

Adhesivity of coating achieved by electroless process with chemical pre-activation was estimated by Vickers indenter. Figures 4a and 4b shows surfaces of specimens that were chemical pre-activated before application of electroless coating. Specimen in Figure 4a was not heat treated, while specimen in Figure 4b was treated by aging at 500°C. It can be seen that delamination of deposited layer was not appeared on both specimens.

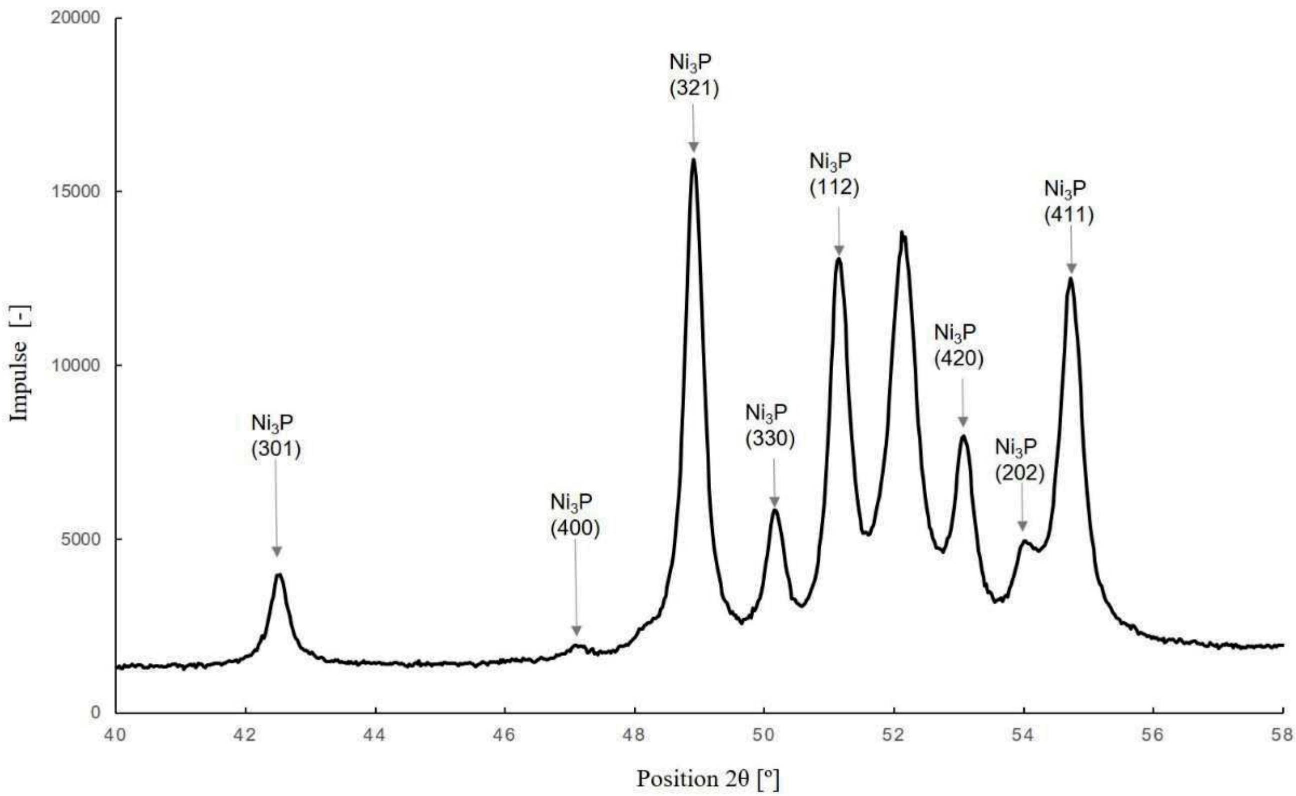

Fig. 3. X-ray diffraction pattern (2θ = 40º-58º) of the heat treated electroless nickel-phosphorous coating showing characteristic Ni_3P reflections with the corresponding Miller indices

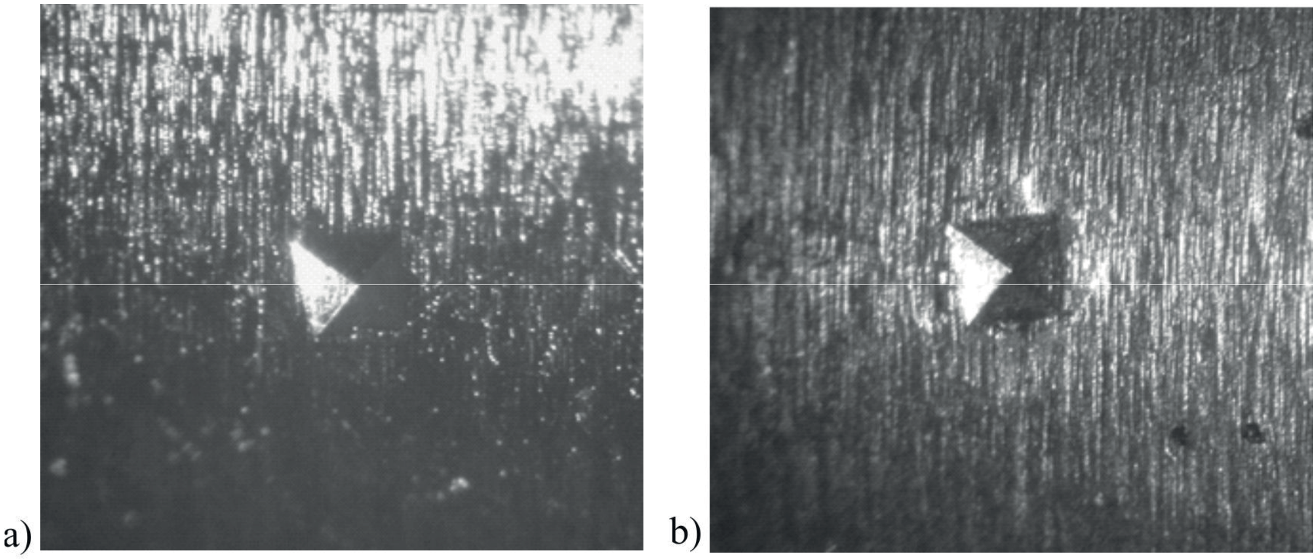

Fig. 4. Indentation results of adhesivity of Ni-P electroless coatings, mag. 35:1, a) chemical pre-coating treatment of surface + electroless coating, b) pre-coating treatment of surface + electroless coating + aging at 500°C

Figure 5 shows the surface pattern after indentation of Vickers indenter on specimen threated by electroless coating without chemical pre-activation. Figure 5 clearly shows areas with delamination of deposited layer. It is evident that the application of an activation process before electroless treatment, increases adhesivity of electroless Ni-P coating.

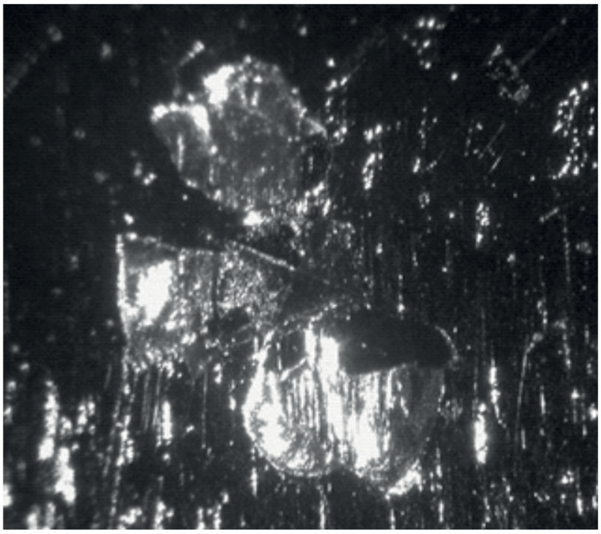

Fig. 5. Indentation results of adhesivity of Ni-P electroless coatings, mag. 35:1, electroless coating + aging at 500°C

4. Conclusions

Applicability of Vickers indenter in testing and optimization of adhesivity of electroless Ni-P coatings deposited on austenitic stainless steel AISI 316 was analyzed.

Surfaces of austenitic stainless steel AISI 316 substrate were activated before depositing the Ni-P coatings by electroless process. Investigated coatings are following the surface morphology of the sample. The uniform Ni-P coatings deposited by electroless process was formed. The thicknesses of Ni-P coating was approximately equal to 8 µm.

Distribution of Ni and P are similar in coating are similar in both, heat treated and non-heat treated specimens.

By X-ray diffraction analysis it was determined, that the Ni_3P phase was formed by heat treatment of samples. At the same time, it was found out that the substantial increase of the hardness of the electroless Ni-P coating is achieved by application of heat treatment.

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 better adhesivity can be achieved.

Vickers indenter can be successfully applied in testing and optimization of electroless Ni-P coatings, deposited on austenitic stainless steel AISI 316.

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