

Investigating Tribological Characteristics of Ni-P and Double-Layered Ni-P/Ni-B Electroless Coatings Applied to the Carbon Mild Steel Ck45

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Abstract

In this research, tribological characteristics of Ni-P electroless coating and double-layered Ni-P/Ni-B electroless coating were studied. For this purpose, Ni-P electroless coating with 25 μm thickness was initially applied to the surface of steel Ck45 samples; then, a double-layered Ni-P/Ni-B electroless coating, including inner layer of Ni-P with 15 μm thickness and outer layer of Ni-B with 10 μm thickness, was applied to the surface of similar samples and were heat treated at 400°C for 1 h. Effect of heat treatment on morphology, structural variations and wear behavior of coating was investigated using different tests such SEM, XRD and pin on disk wear test at room temperature. Hardness of the obtained coatings was measured by a Vickers microhardness machine prior and after heat treatment of the samples. The results showed that the double-layer coating with the same thickness in each layer improved hardness and wear resistance compared to the single-layer coating. Heat treatment led also to the creation of nano crystalline structure; therefore, hardness and wear resistance of the coating increased due to change in structure from amorphous to crystalline structure and created rigid phases of Ni₃P.

Keywords: Double-layer electroless coating, Heat treatment, Nano crystalline structure, Hardness, Wear resistance.

1. Introduction

Electroless coating method is widely applied to nickel based coating. In this method, a metal is deposited by the reduction of metallic ions available in the bath using a reducing factor unless it is applied to an external current. This coating has uniform thickness and excellent properties such as high corrosion resistance, good wear resistance and non-magnetic characteristics due to its manufacturing process and individual composition¹⁾. Recently, electroless nickel plating bath with boron hydride agent has been considered. Properties of Ni-B electroless coating is often similar to that of Ni-P electroless coating. The main advantage of Ni-B electroless coating is excellence in hardness and wear resistance²⁾. Also, applying heat treatment to nickel electroless coatings forms nanocrystalline grains and rigid phases in the coating; as a result, hardness and wear resistance are increased^{3,4)}. The aim of the present study was to create a double-layered Ni-P/Ni-B electroless coating

and investigate effect of heat treatment on hardness and wear resistance of the formed coating.

2. Materials and methods

Disc samples with 5 mm thickness and 50 mm diameter were prepared from steel Ck45 and sand blasted by silicon particles with mean size of 100 μm . Sand blasting the samples was conducted prior to the coating step in order to increase coating adhesion to the substrate. After sand blast operation, the samples were coated by Ni-P and double-layered Ni-P/Ni-B electroless coating. A commercial electroless solution (SLOTONIP70) made by German Schloetter Co. was applied. PH 4.6 for plating bath and 85°C as coating temperature were considered. Chemical composition of the plating bath is reported in Table 1. Coating operation was conducted at 85°C and pH 12.5 in order to create Ni-B electroless coating. Then, some coated samples were heat treated for 1 h at 400°C in an inert atmosphere. A scanning electron microscope was applied in order to study morphology of the samples' surface and X-Ray diffractometry was used for their phase analysis. Micro-hardness of the samples was measured by the initial loading of 100 g in Vickers scale. In order to improve accuracy of micro-hardness test results, this test was repeated 5 times for similar samples and the mean number was reported as microhardness result. Wear test was conducted using

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pin on disk method in order to evaluate tribological behavior of the samples. The pin applied to wear tests was made of steel 52100 with hardness of 65 HRC. Wear test was conducted by loading 5 N at speed 0.1 m/s for maximum travel distance of 1000 m at ambient air.

Table 1. Chemical composition of Ni-B electroless plating bath

Material	Concentration (g/lit)
Nickel chloride	20
Sodium boron hydride	1
Ethylene diamine	56
Sodium hydroxide	40
Lead nitride (Pb^{2+})	0.01

3. Result and discussion

3.1. Study on the morphology and the cross-section of coatings

Morphologies of double-layer Ni-P/Ni-B electroless coating and Ni-P electroless coating applied to the steel substrate are shown in Figures 1(a) and 1(b), respectively. Figure 1 shows that morphology of the created coating was cauliflower which was quite common in Ni-P and Ni-B electroless coatings^{1,2}.

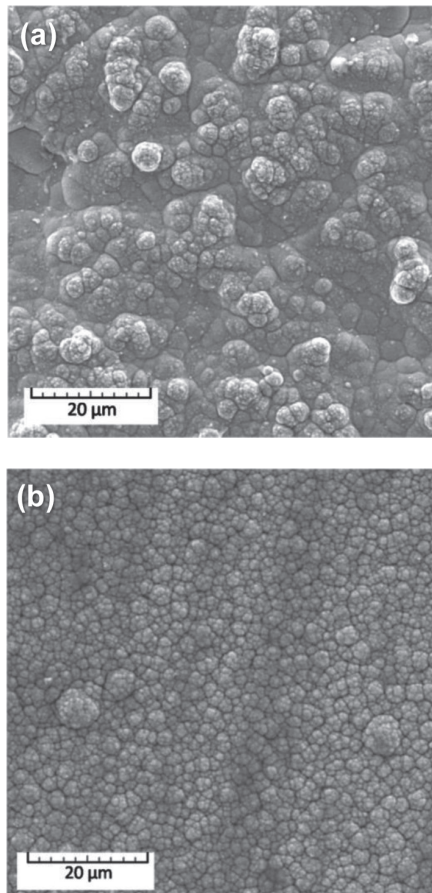


Fig. 1. SEM images of coating surface: (a) Ni-P/Ni-B and (b) Ni-P.

The traverse sections of Ni-P electroless coating and double-layered Ni-P/Ni-B electroless coating are given in Figure 2. As is obvious in this figure, a uniform coating was formed on the substrates and a desirable adhesion was established between the coating and substrate. Thickness of Ni-P layer in single-layered coating was about 25 μm . Thickness of different layers in the double-layer coating was about 15 μm in Ni-P layer and about 10 μm in Ni-B layer.

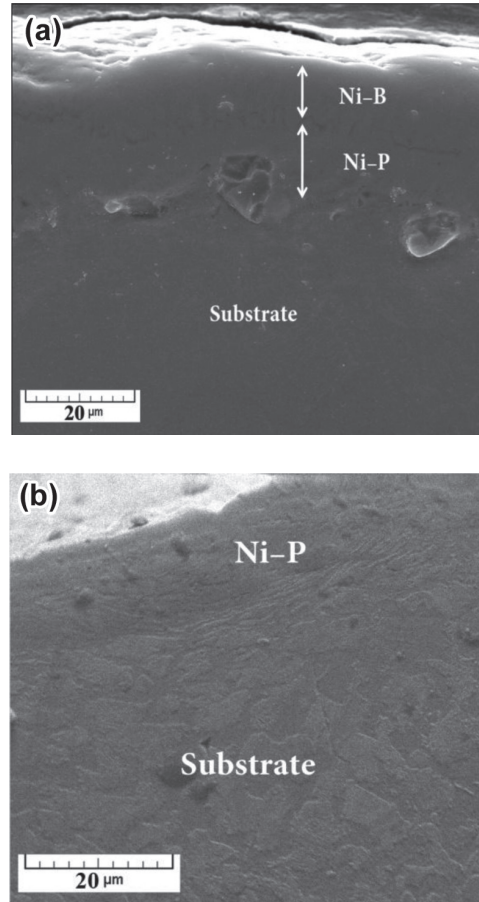


Fig. 2. Cross-section SEM images of: (a) double-layered Ni-P/Ni-B and (b) Ni-P electroless coating.

3.2. Effect of heat treatment on coatings structure

X-Ray diffraction patterns of Ni-P electroless coating and double-layered Ni-P/Ni-B electroless coating in two states of prior and after heat treatment at 400°C for 1 h are shown in Figures 3 and 4. As represented in these figures, electroless coatings contained a mixture of amorphous and crystalline structure prior to heat treatment; i.e. distinct peaks from two phases of crystalline nickel with FCC lattice and Ni_3B/Ni_3P phase with orthorhombic lattice were observed in diffraction diagrams instead of amorphous peaks^{3,4}. The research showed that minimum temperature required to the transformation of amorphous to crystalline structure in Ni-B and Ni-P

electroless coating was about 300-350 °C and increase in heat treatment temperature led to more crystallized Ni-P and Ni-B electroless coatings measured by X'pert high score, version 1.0 d, software using Scherrer method⁵). The result showed that heat treatment caused crystallization of the structure in the coatings.

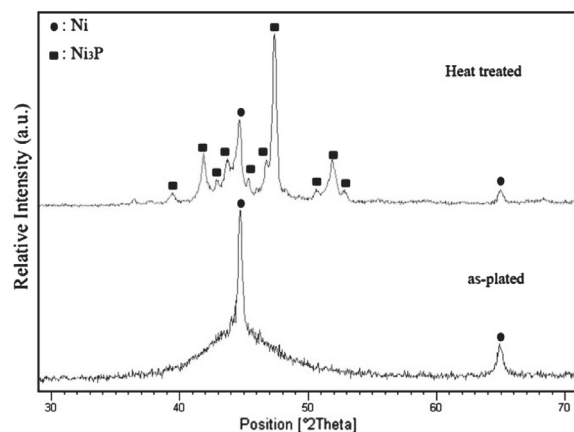


Fig. 3. X-Ray diffraction pattern of Ni-P electroless coating prior and after heat treatment at 400 °C for 1 h.

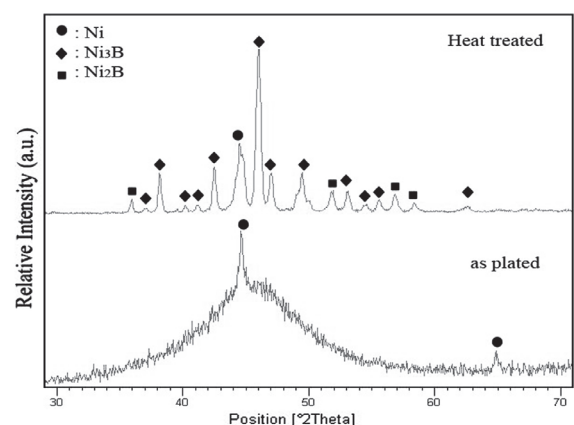


Fig. 4. X-Ray diffraction pattern of double-layered Ni-p/Ni-B electroless coating prior and after heat treatment at 400 °C for 1 h.

3.3. Effect of heat treatment on coatings hardness

Hardness of Ni-P and double-layered Ni-p/Ni-B electroless coatings prior and after heat treatment is represented in Table 2. As can be seen in this table, hardness of coatings intensively increased after heat treatment. The research showed that increase in hardness was related to precipitation of stable intermetallic compounds such as Ni₃P and Ni₃B during the crystallization of amorphous phase^{3,4}). It was reported that high strength and shear modulus for Ni₃B phase affected hardness⁶). These observations were in suitable agreement with results of XRD analysis.

Table 2. Hardness of the created coating prior and after heat treatment

Coating	Condition	Hardness (Hv)
Ni-P	Non-heat treated	600
Ni-P	Heat treated at 400 °C	973
Ni-P/Ni-B	Non-heat treated	946
Ni-P/Ni-B	Heat treated at 400 °C	1427

3.4. Effect of heat treatment on wear behavior of coatings

Wear tests were conducted under the loading 5 N based on load wearing capacity test. The diagrams of weight loss versus travel distance at ambient air are shown in Figure 5. As can be seen, heat treated samples at 400 °C had the minimum weight loss and, on the other hand, non-heat treated samples had the maximum weight loss. Decrease in weight loss of the heat treated samples was related to the precipitation of stable Ni₃P phases during the crystallization of amorphous phase which was provable with respect to the results of XRD patterns and micro-hardness test^{7,8}).

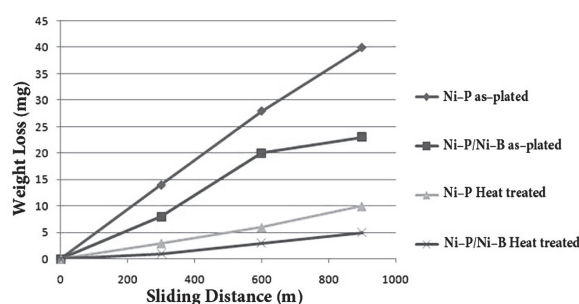


Fig. 5. Diagrams of weight loss versus sliding distance.

SEM images of wear surfaces are shown in Figure 6. As can be seen in Figures 6(a) and 6(c), worn surface of the sample had low hardness prior to heat treatment because no crystalline phase was formed; this led to severe plastic deformation, formation of edge in the coating as a result of plastic deformation, formation of local joints in contact areas and rupture of these joints during sliding process. So, the dominant mechanism was adhesive wear^{7,8}). Figures 6(b) and 6(d) show that heat treatment at 400 °C for 1 h led to transformation of amorphous structure to crystallized nickel and formation of Ni₃B and Ni₃P and these phases improved wear resistance. SEM images of the samples represented that coating of the samples was maintained on the surface, even after sliding distance of 1000 m. These observations were in agreement with microhardness, XRD and weight loss results.

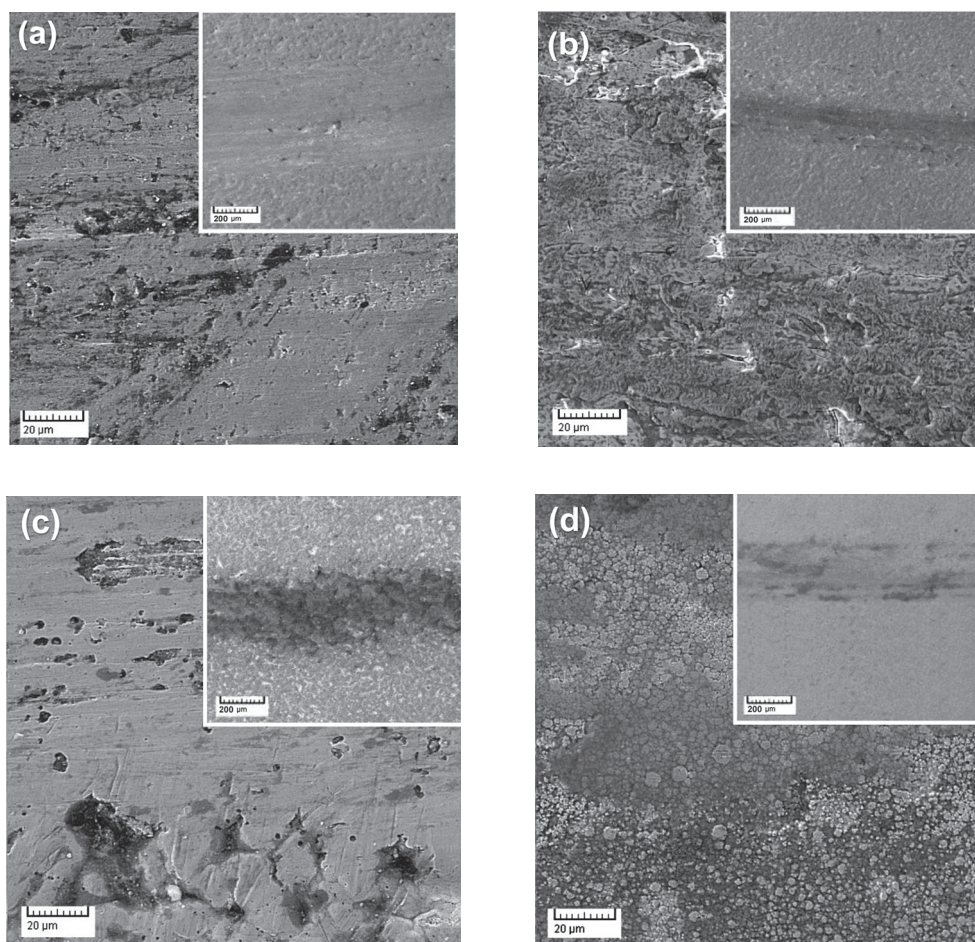


Fig. 6. SEM image of worn surfaces of electroless coatings: (a) non-heat treated Ni-P, (b) heat treated Ni-P, (c) non-heat treated double-layered Ni-P/Ni-B and (d) heat treated double-layered Ni-P/Ni-B.

4. Conclusion

1. Morphology of Ni-P electroless coating and double-layer Ni-P/Ni-B electroless coating was semi-amorphous with a cauliflower structure.
2. Hardness of double-layered Ni-P/Ni-B electroless coating was more than that of Ni-P electroless coating with the same thickness.
3. Hardness of coating was intensively increased after heat treatment. This increase was related to precipitation of stable Ni_3B and Ni_3P intermetallic compounds during the crystallization of amorphous phase.
4. Heat treatment at 400°C caused formation of a nano-crystalline structure; therefore, hardness and wear resistance of the coating increased because of transformation from amorphous structure to crystalline structure and formation of rigid Ni_3P and Ni_3B phases.
5. According to SEM images and weight loss amounts, double-layered Ni-P/Ni-B electroless coating heat treated at 400°C for 1 h had the highest wear resistance.

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