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Study of the Effect of Heat Treatment and Layer Thickness on the Hardness of Chemically Heated Nickel Coating

Alikabulova Nafisa Xolmirza qizi

Jizzakh polytechnic institute, assistant teacher

Abstract: *This study aims to analyze the effect of heat treatment on the phase transformation of a thin nickel layer on a steel plate substrate. The plating process is electroless plating using a chemical solution. This work requires a nickel solution as the main component of the coating. The substrate was electroless nickel plated at pH 7.5 and the temperature was 85℃ for 100 minutes with heat treatment at 320℃ and 340℃ for 30 minutes. In addition, the substrate is analyzed by metallography to obtain a visual representation of its thickness on the surface of the plate. The result shows that the layer on the steel surface has the same thickness. Based on the hardness test, the heated plate resulted in better performance compared to the unheated one. The surface hardness increased from 134 to 646 with a coating thickness of 17.49 mm, and the XRD characteristic showed a broadened Ni peak. After heat treatment at 320℃ and 340℃, the surface hardness increased to 851VHN and 1020VHN.*

Keywords: *electroless plating; heat treatment; hardness; nickel.*

Introduction: The equipment components used in many industrial companies are usually steel materials. Many components cause surface friction during their use and cause component wear. Material wear, especially steel, shortens component life. In addition, steel components are prone to corrosion. Therefore, we need to protect steel parts against wear and corrosion. Surface engineering technology or surface engineering is commonly used in industry to optimize the performance of equipment. Surface engineering generally focuses on improving the hardness and corrosion resistance of parts that rub against each other and producing durable parts for industrial tools and equipment. One of the methods used is electroplating.

At the same time, the electroplating method is prone to hydrogen gaps, and it is difficult to form uniform layers, especially at the edge of the sample. Electroless Nickel Plating (ENP) is a nickel plating process on the surface of an electroless substrate. Electroless nickel is an alternative method to prevent hydrogen embrittlement. It also produces a layer of uniform thickness and good corrosion resistance. The application of electroless nickel is limited by the size of the component to be plated. In order to obtain the maximum hardness value, studies were conducted on the effect of parameters on electroless nickel. These parameters include solution composition, solution pH, temperature, and time.

After making electroless nickel, heat treatment is also done to increase the hardness of the coating. During heat treatment, temperature and time affect the final hardness of the layer. Electroless nickel ions did not show the presence of Ni and.

Ni-P compounds by XRD characterization. After heat treatment, crystalline Ni and Ni3P appear in the XRD results. Ni3P compounds appear after heating at 400 ℃. Therefore, it is necessary to study some parameters that affect the mechanism of hardening. In this study, the phase formed in the ENP layer was analyzed to determine the reason for the increase in hardness after heat treatment.

1. Methodology

Usually, a surface layer is coated before the workpiece is used in various ways to obtain the desired properties. A coating or coating is a surface layer that comes from a material different from the coating material (substrate). This study used an electroless plating method to deposit nickel on a steel plate. Electroless plating is a plating method that uses chemical reactions without the need for electricity. Electroless nickel plating (ENP) is one of the most common plating methods using nickel as the plating material. The phenomenon of precipitation of nickel as a result of the reflux of salt solution was discovered by Waltz in 1844. The electroless nickel plating method is widely used because it has several advantages.

1. The nickel layer produced by ENP is usually the same on the entire surface of the outer and inner substrate, so it can be used to cover the machining part with complex shapes. In addition, ENP is a good choice for coating because of its corrosion resistance due to the uniform coating layer, resulting in a good coating for the substrate surface. The electroless nickel method is more efficient because it does not require complex equipment and the end result is a better finishing process.

1.1. Specimen preparation: The specimens used are low carbon steel plates with a length of 30, a width of 15 mm and a thickness of 0.5 mm. The surface to be is first sanded and polished until smooth. Then, the surface is washed and washed with an acetone solution. The used solution is after 1 mixture for coating. The ingredients used are mixed with the composition as in table 1. The chemical solution is a mixture of several chemical compounds and works on electroless nickel. The solution used contains a source of nickel, a reducing agent, a complexing agent, an incubator and a buffer

1.2. Coating process and testing

The coating process is carried out by soaking into the steel plate, the temperature of the solution for about 100 minutes, the solution is kept constant at 85℃ during the coating process. As well as temperature, pH 7.5 is kept constant during the process, sodium hydroxide is added. Layers are formed due to autocatalysis.

Chemical reactions. Reduction of nickel ion is carried out using hypophosphite compounds. The initial deposit becomes a catalyst.

The reduction reaction itself. The chemical reaction occurs continuously, as a result of which the thickness of the coating layer increases, the autocatalytic reaction, and the substrate does not decrease, because the oxidation reaction after the reduction reaction is a hydrogen evolution reaction.

After coating, the sample is heat treated in a furnace with a.

Hold time 30 minutes at temperatures of 320 ℃ and 340 ℃. The hardness test was performed using the micro Vickers method. 0.05 kg load. X-ray diffraction (XRD) effect was carried out. To determine the effect of heat treatment on the structure, a nickel layer is formed. Monitoring of the quality of the coating layer and thickness measurements are carried out by the method of metallography.

Table 1: Solution composition

Compound	Chemical Formula	Qty. (g/L)	Phase
Nickel chloride	$NiCl_2.6H_2O$	45	Solid
Sodium hypophos-phite	$NaH_2PO_2.H_2O$	11	Solid
Ammonium chloride	NH_4Cl	50	Solid
Sodium citrate	$Na_3C_6H_5O_7.2H_2O$	100	Solid
Sodium hydroxide	NaOH	= pH 7,5	Liquid

Based on the metallography depicted in Figure 1, electroless nickel forms a uniform deposition layer on the surface of the steel sheet. The average thickness of the deposited layer is 17.9 µm. The microstructure of pearlite and ferrite in the substrate indicates the steel plate used.

Microstructure of nickel

The layer is not visible in the metallographic results. The results of the conducted studies show that its microstructure is amorphous, crystalline, or both. However, the composition of the phosphorus element in the electroless nickel ion determines the microstructure of the ion and the nature of the coating.

Low phosphorus kions, 1-5% by weight, gives a crystalline microstructure. Deposits with medium and high phosphorus content, 6-9% by weight and 10-13% by weight, the microstructure is amorphous and crystalline. Its properties are strongly influenced by the electroless nickel layer microlayer. The thickness of the layer is very small compared to the sample thickness of 0.5 mm. However, a thin layer can lead to a relatively large increase in surface hardness. This shows that the layer has a higher hardness than the substrate. Hardness of steel plate 134 before plating.

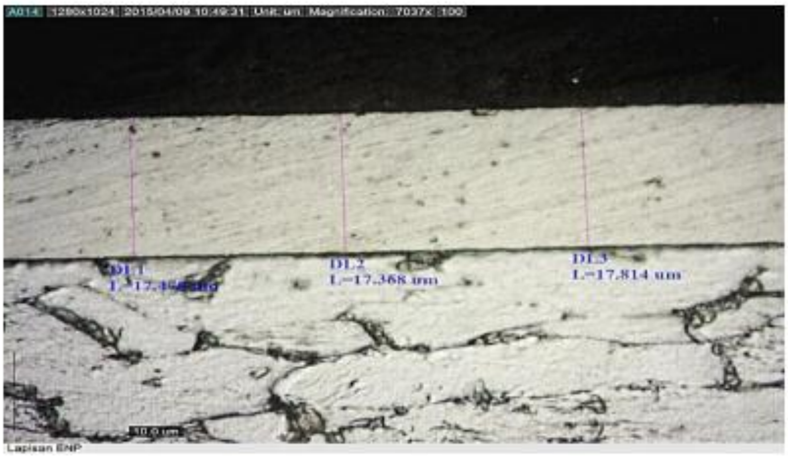


Figure 1: Metallography of the sample cross-section

Table 2: Hardness test result

Indentation	Before Coting (VHN)	After Coating (VHN)	HT 320°C (VHN)	HT 340°C (VHN)
1	137	584	824	1049
2	124	689	780	927
3	140	644	927	965
4	133	677	841	985
5	139	613	985	1072
6	135	644	766	1072
7	128	677	946	1049
8	135	655	810	985
9	137	655	841	1027
10	139	623	795	1072
Avg.	134	646	851	1020

As shown in Table 2, the initial surface hardness of the substrate is 134. After electroless nickel plating, the surface hardness increases to 646. Surface hardness increases after electroless nickel coating. The increase in hardness is due to the deposition of an electroless nickel coating, which is a Ni-P alloy. Based on the Ni-P equilibrium phase diagram, the freezing process produces cryst.

Pure Ni and Ni3P compounds. Ni3P is a compound between a metal and a non-metallic ceramic, so it is hard. However, as shown in Figure 2, the XRD results after coating do not. indicates the presence of Ni and Ni3P. In XRD, only one result appeared with peak broadening. Compared with the diffraction patterns of Fe and Ni, the peak is more consistent as Ni. peak. The broadening of the XRD peak from the sample can be caused by the presence of an amorphous phase, the inhomogeneity of the unit cell, and non-uniform voltage. The electroless nickel deposition process is different from the metal solidification process.

During solidification, Ni3P compounds are formed at a temperature of 880 °C. Although electroless nickel occurs at a much lower temperature and in a relatively short time, so Ni3P does not have time to form. Ni deposits may not be able to organize themselves as crystals.

Ni unit cell FCC (face centered cubic). To form a crystalline structure, these unit cells are neatly arranged with a specific direction. In electroless nickel, the cells of these unit cells are not clean, and the unit layer continues to grow thick, because the organized cell is not yet arranged. As a result, the deposit layer is amorphous. The nature of this amorphous layer is similar to solid glass.

Surface hardness increased after heat treatment for 30 minutes.

As shown in Table 2, the hardness after heat treatment at 320°C is 851. At the same time, heat treatment at a temperature of 340 °C is given.

Hardness 1020 is due to increased hardness,

A transformation from an amorphous ion structure to a crystalline one has been demonstrated Sharp peaks of Ni and Ni3P in XRD results.

Summary. (FWHM), α and k subscript for amorphous and crystalline.

The FWHM value is measured using XPowder software. Heat Treatment at 320 °C resulted in 69.9% amorphous crystalline transformation and at 340 °C 75.3%. This is a change Corresponds to the increased hardness test result of 851VHN and 1020VHN

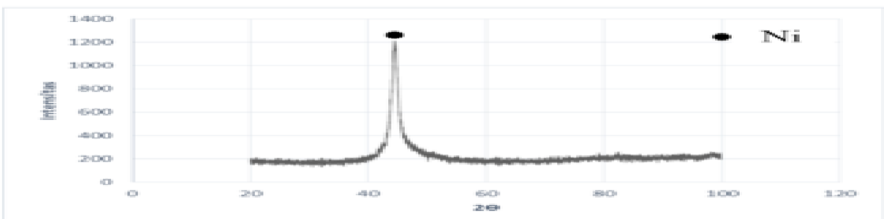


Fig. 2: XRD result of coated specimen

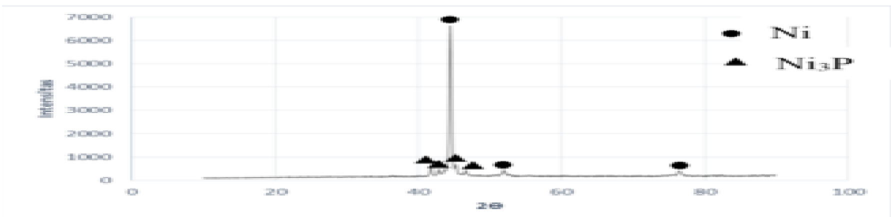
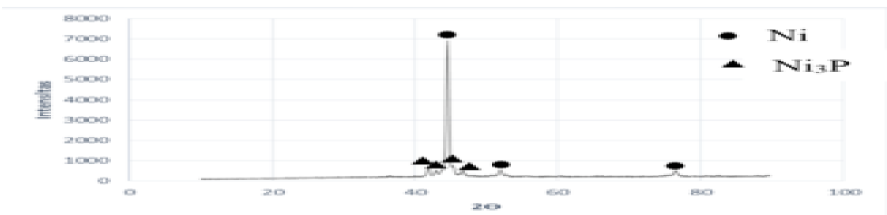


Fig. 3: XRD result of specimen after HT 340°C for 30 minutes



Summary.

Heating at 320℃ and 340℃ for 30 minutes transformed the amorphous Ni-P coating layer into crystalline structures.

by increasing the hardness to 851VHN and 1020VHN. Justified

The XRD results showed that crystalline Ni and Ni3P peaks appeared after heating.

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