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Research paper

The Heat Treatment effect of Nickel-coated Steel Plate Substrate by Electroless Plating on Hardness Test

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Abstract

The present study was aimed to analyze the effect of heat treatment on phase change of thin layer of nickel on steel plate substrate. The coating process was electroless plated using chemical solution. This work requires nickel solution as the main component of coating. Substrate was coated by electroless nickel plating at pH 7.5 and temperature was 85 ℃ for 100 minutes with heat treatment for 30 minutes at temperature are 320 ℃ and 340 ℃. Furthermore, the substrate will be analyzed with metallography method to obtain visualization of thickness on plate surface. The result show that the layer on steel surface has a uniform thickness. Based on the hardness test, the heated plated resulting the better performance compared with the non-heated one. Surface hardness increased from 134VHN to 646VHN with a coating thickness of 17.49μm and a XRD characterization showed a broadened Ni peak. After heat treatment at temperature are 320℃ and 340℃, the surface hardness increased up to 851VHN and 1020VHN.

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

1. Introduction

In many industry companies, components of equipment used are generally steel materials. Many components will experience surface friction in their use and will cause the component to wear. Wear of the material, especially steel will reduce the life time of the component. In addition, components from steels are susceptible to corrosion. Therefore, we need a wear protection and corrosion against steel components.

Surface engineering technology or is called as surface engineering is commonly used to optimize the performance of equipment in industry. Surface engineering generally is aimed to increase the hardness and corrosion resistance of the components that rub against each other and produce the durable components for industrial tool and equipment. One method used is electroplating. However, the electroplating method is susceptible to hydrogen embtittlement and it is difficult to produce uniform layers, especially at the edge of the specimen.

Electroless nickel plating (ENP) is a nickel coating process on the surface of the substrate without electricity. Electroless nickel is an alternative method to avoid hydrogen embrittlement. It also produces layer with uniform thickness and good corrosion resistance. The application of electroless nickel is limited to the size of the component to be coated [3].

Research was conducted on the effect of the parameters on electroless nickel to get the maximum hardness value. These parameters include the composition of the solution, pH of the solution, temperature, and time. Heat treatment after performing electroless nickel is also done to increase the hardness of the coating. Temperature and time in the heat treatment process affect to the final hardness of the layer [4].

Electroless nickel deposits did not show the presence of Ni and Ni-P compounds through XRD characterization [2]. After heat treatment, the crystalline Ni and Ni_3P appear on XRD results [6-7]. Ni_3P compounds appear after heating at a temperature of 400℃. Therefore, it is necessary to investigate some parameter that affect to the mechanism of increasing the hardness. In the present study, the phase formed in the layer was analyzed to determine the cause of the increase in hardness after heat treatment in the ENP layer.

2. Methodology

Generally, the surface layer is coated before the workpiece is used with various methods to have the desired properties. Coating or plating is a surface layer that comes from a material that is different from the coated material (substrate). The present study was using electroless plating method for nickel deposition on steel plate. Electroless plating is a coating method through chemical reactions without electricity needed. Electroless nickel plating (ENP) is one of the commonly used coating methods, using nickel as a coating material. Nickel deposition phenomenon from the reduction of salt solution was discovered by Waltz in 1844.

Electroless nickel plating method is widely used because it has several advantages. The nickel layer produced by ENP is commonly uniform on entire substrate surfaces, both external and internal, so that it can be used for coating workpiece with complex shapes. Furthermore, ENP is good choice for coating due to its durability on corrosion caused by uniform coating layer resulting good cover for substrate surface. The electroless nickel method is more efficient because it does not require complicated equipment and the final result is good without finishing process [5].

2.1. Specimen preparation

The specimens used are low carbon steel plates with a length of 30 mm, width of 15 mm, and thickness of 0.5 mm. The surface that will be coated is firstly grinded and polished until smooth. Then, the surface is washed and rinsed with aceton solution. The solution used for coating follows a mixture of bath 1 [1]. The ingredients used are mixed with the composition as in Table 1. The chemical solution used is a mixture of several chemical compounds with their own functions in electroless nickel. The solution used contains source of nickel, reducing agent, complexing agent, inhibitor, and buffering agent.

2.2. Coating process and testing

The coating process is carried out by soaking the steel plate into the solution for approximately 100 minutes. The temperature of the solution was kept constant at 85℃ during the coating process. As well as temperature, pH 7.5 is also kept constant during process by adding sodium hydroxide. Layers are formed due to autocatalytic chemical reactions. Nickel ion reduction is carried out by hypophosphite compounds. The initial deposit becomes the catalyst of the reduction reaction itself. The chemical reaction will occur continuously resulting the increasing in thickness of the coating layer due to autocatalytic reaction, while the substrate does not decrease because the oxidation reaction that follows the reduction reaction is the hydrogen evolution reaction.
After being coated, the specimen is heat treated in a furnace with a holding time of 30 minutes at temperatures of 320 ℃ and 340℃. Hardness test was carried out using the micro Vickers method with 0.05 kg load. X-ray diffraction (XRD) characterization was carried out to determine the effect of heat treatment on the structure of the nickel layer formed. Coating layer quality observations and thickness measurements are carried out by metallography method [6].

Table 1: Solution composition

Compound	Chemical Formula	Qty. (g/L)	Phase
Nickel chloride	$NiCl_2.6H_2O$	45	Solid
Sodium hypophosphite	$NaH_2PO_2.H_2O$	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

3. Results and Discussion

Based on metallography as described at Fig. 1, the electroless nickel produces the uniform deposite layer on steel plate surface. The average thickness of deposited layer was 17.9µm. The microstructure of perlite and ferrite on the substrate shows the steel plate used is a low carbon steel material. The microstructure of the nickel layer is not seen in metallographic results. The results of the research that have been carried out show that the microstructure of electroless nickel deposits is amorphous, crystalline, or both. However, the content of the phosphorous element in the nickel electroless deposit will determine the microstructure of the deposit and the nature of the coating. Low phosphorous deposits, 1–5 wt%, gives crystalline microstructure. While deposits with medium to high phosphorus content, 6-9 wt% and 10-13 wt%, the microstructure produced is amorphous and crystalline [9]. The properties of the electroless nickel layer are strongly influenced by the microstructure layer.
The thickness of the layer is very small when compared with a sample thickness of 0.5mm. But, the thin layer can produce a relatively large increment in surface hardness. This shows that the layer has a higher hardness than the substrate. The hardness of the steel plate before being coated is 134VHN [7].

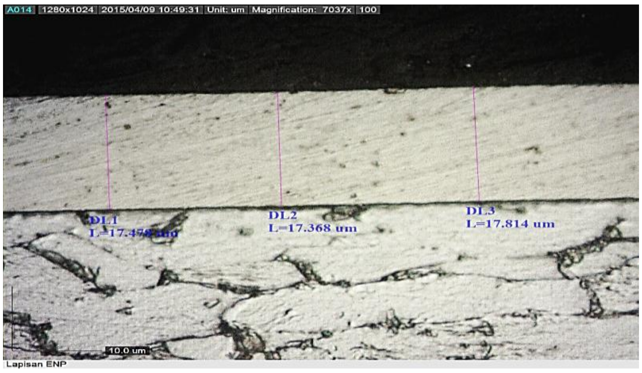

Fig. 1: Metallography of specimen cross section

Table 2: Hardness test result

Indentation	Before Coting (VHN)	After Coating (VHN)	HT 320℃ (VHN)	HT 340℃ (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 134VHN. After elecroless nickel plating, surface hardness increase to 646VHN. Surface hardness increases after electroless nickel plating. This increasing in hardness is due to the deposit of electroless nickel plating, which is a Ni-P alloy. Based on the Ni-P equilibrium phase diagram, the freezing process produces crystalline pure Ni and Ni_3P compounds. Ni_3P is a compound between metal and non-metal which is a ceramic, so it is hard.
However, XRD results after coating as shown in Fig. 2 do not indicate the presence of Ni and Ni3P. On the XRD results only one peak appeared with the peak broadening. When compared to the diffraction pattern of Fe and Ni, the peak is more suitable as Ni peak. Widening of the XRD peak from the sample can be caused by the presence of amorphous phase, unit cell non-homogeneity, and non-uniform strain.
The deposition process of electroless nickel itself is different from the metal solidification process. In the solidification process, Ni_3P compounds are formed at a temperature of 880℃. While electroless nickel takes place at a much lower temperature and a relatively short time, so Ni_3P does not have time to form. Ni deposits themselves may not be able to organize themselves as crystalline Ni [9].
Ni unit cell is FCC (face center cubic). To form a crystalline structure, these unit cells are neatly arranged with a specific orientation. In electroless nickel, the cells of these unit cells are not neatly arranged because the layer continues to grow thicker while the unit cell has not yet been arranged. As a result, the deposit layer is amorphous. The nature of this amorphous layer is like hard glass.
Surface hardness increased after heat treatment for 30 minutes. As seen in Table 2, the hardness after the heat treatment at 320℃ is 851VHN. While, heat treatment at a temperature of 340℃ gives hardness 1020VHN. The increasing in hardness occurred due to changes in amorphous deposit structure to crystalline proven by the sharp peaks of Ni and Ni_3P in the XRD results Fig. 3 and Fig. 4.
During heat treatment, atoms diffusion can occur because the diffusion process is a function of temperature and time. So that, amorphous deposits are able to organize themselves into orderly arrangement. Amorphous-crystalline changes are calculated using the formula $\frac{B_K \times I}{B_a \times I + B_K \times I}$ where B is Full Width at Half Maximum

(FWHM), subscripts a and k for amorphous and crystalline. The FWHM value is measured using the XPowder software. Heat treatment at a temperature of 320℃ resulted in an amorphous-crystalline change of 69.9% and at 340℃ of 75.3%. This change is in line with the increase in hardness test result, which is 851VHN and 1020VHN [9].

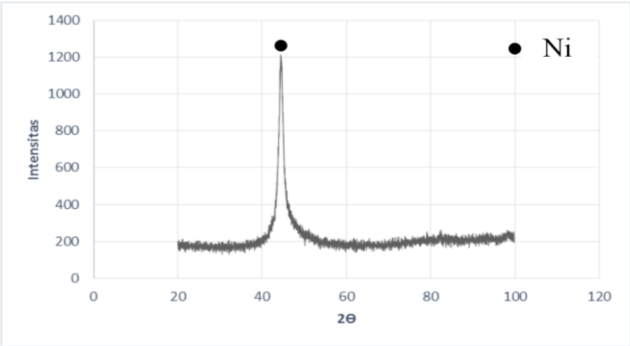

Fig. 2: XRD result of coated specimen

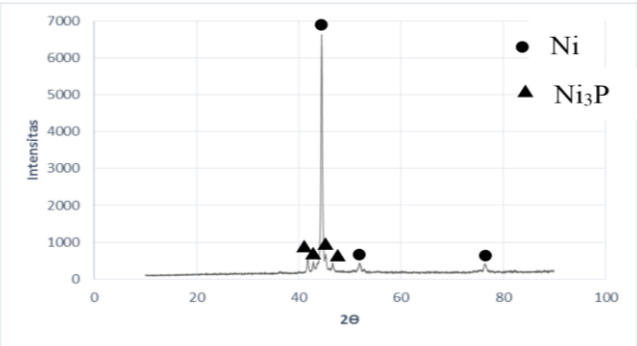

Fig. 3: XRD result of specimen after HT 340℃ for 30 minutes

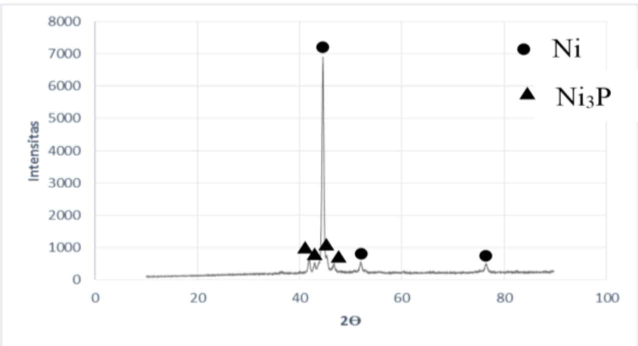

Fig. 4: XRD result of specimen after HT 340℃ for 30 minutes

4. Conclusion

Heating at 320℃ and 340℃ for 30 minutes transformed amorphous Ni-P coating layer into crystalline structures characterized by increasing in hardness to 851VHN and 1020VHN. Based on XRD results after heating appeared crystalline Ni and Ni_3P peaks [10].

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