CORROSION BEHAVIOR OF Ni THIN FILM PLATING

Nor Akmal Fadil[1,2], K. Noda[1], Muhammad Zaimi[1], Atuty Amrin[2]
[1] Department of Materials Science and Engineering, Shibaura Institute of Technology
3-7-5 Toyosu, Koto-ku, Tokyo 135-8548, Japan
[2] Department of Materials and Engineering, Universiti Teknologi Malaysia, 81310 Skudai, Johor Bahru, Malaysia.

Introduction

This study is related to investigation of corrosion behavior of Nickel and its alloys of thin film in corrosive environment in chemical and oil and gas industries. More particularly it is related to the application of anodes in impressed current cathodic protection system.

An impressed current anode should have an ability to deliver high current supply as possible and usually have a more positive potential than the protected object. Other than that, it should have low solubility as possible, high impact resistance and not to be damaged by abrasion or vibration. An impressed current anode also should have high conductivity and be capable of heavy loads.

Existed anode available for ICCP, a mixed metal oxide have very low consumption rates, better mechanical properties as compared to the brittle iron base anode [1] and cheaper than platinized anode. But, the manufacturing process of mixed metal oxide anodes is difficult because they demand complex process of preparations which may cost them relatively higher than other commercial anodes [2]. Thus, the less expensive deposition method for nickel base alloy plating on pure iron substrate was studied which is electroless Ni-Co-P.

Experimental

In this study, a sample 99% Fe substrate used with the dimension of 15 x 15 x 0.5 mm. The substrates were subjected to a series of pretreatment as preparing the samples for electroless plating process. A mixture of stannum chloride and HCl solution used as surface sensitization while for the surface activation, the mixture of palladium chloride and HCl is used. After the pretreatment process, the catalyzed surfaces of substrates then were subjected to electroless Ni-Co plating. The components and specification of plating solution are shown in the Table 1.

Table 1: Plating Bath Components and Operating Parameters

$NiSO_4 \cdot 6H_2O$ (M)	0.045	
$CoSO_4 \cdot 7H_2O$ (M)	0.005	
$Na_3C_6H_5O_7 \cdot 2H_2O$ (M)	0.15	(complexion agent)
$NaH_2PO_4 \cdot H_2O$ (M)	0.30	(reducing agent)
NH_4Cl (M)	0.50	(buffering agent)
pH	4.5, 9	
Temperature (°C)	70	
Plating time (seconds)	3600	

The pH of the solution is adjusted by using sodium hydroxide for obtaining alkaline solution and sulfuric acid for acidic solution. Two sets of bath solution were prepared for different pH with the temperature of 70^0C for several periods of times. The temperature for the bath is critical in obtaining a uniform and consistent thickness of nickel layer. In order to investigate the effect of activation and sensitization processes on deposition rate, another set of samples were run using the same method but with the absent of activation agent during the pretreatment process.

A cyclic potentiodynamic polarization measurement was conducted in order to determine susceptibility to localized corrosion in an aqueous solution of 0.05M NaCl and maintained at a constant room temperature. The control electrode and reference electrode was platinum and Ag/AgCl/kCl saturated respectively. The plating layer and corrosion products were characterized by using X-ray Diffraction (XRD) and their surface morphology were examined by Scanning Electron Microscopy (SEM).

Results and Discussion

From the electroless plating study, the deposition rate is measured and analyzed. The results show that deposition rate for Pd-activated and Sn-sensitized substrates have similar result as compared to the samples without those processes. This shows that the activation and sensitization processes did not influent the deposition rate. However the alkaline solution which higher value of pH possess higher deposition rate as compared to acidic bath solution.
The X-ray diffraction pattern of electroless Ni-Co-P coatings obtained. The most intense reflection in Pd-

activated and Sn-sensitized samples represents Fe-Co alloys while the coating layers of samples without activation and sensitization processes exhibit Ni-Co-P alloys. The surface morphology observation done by SEM shows that deposition Ni-Co-P thin film without activation and sensitization formed relatively more uniform coating (Figure 1) with high phosphorous content that is confirmed by the EDAX analysis as compared to activated and sensitized samples.

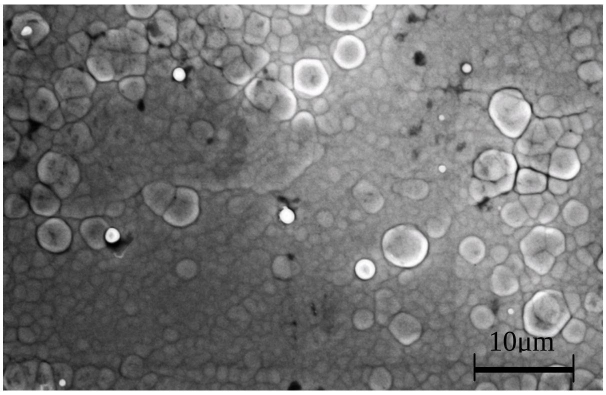

Figure 1: SEM micrograph of electroless Ni-Co-P coating surface without activation and sensitization.

The obtained results from the electrochemical potentiodynamic anodic measurement are shown by the graph in Figure 2. These results show a comparison in pitting and passivation behavior between un-activated and Pd-activated deposition in 0.05M concentration of NaCl.

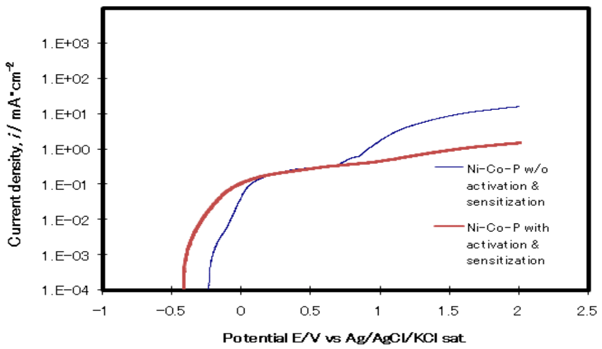

Figure 2: Results of anodic polarization curves for deposition in alkaline solution.

The polarization curves from Figure 2 show that corrosion and pitting potential for the deposition without activation and sensitization in alkaline solution is higher than those were subjected to activation and sensitization processes. The higher value in corrosion potential is associated with the presence of high phosphorous content in Ni-Co-P coating layer [3]. While lower corrosion potential of activated and sensitized samples are associated with lower resistance to pitting corrosion and the presence of Cl^- increased localized attack in the form of pitting and crevice attack as well. The passive film starts to form at relatively lower current density for the un-activated and sensitized coating which is about 1.5 $x10^{-3}$ mA/cm^2 before this protective layer were destroyed by pitting at corrosion potential of -0.1 V vs Ag/AgCl/KCl sat. However the second phase of passive layer formation slightly higher for this sample as compared to those subjected to the activation and sensitization after the second pitting attacked at 0.6 V vs Ag/AgCl/KCl sat.

If this finding is compares to corrosion behavior of pure nickel in the previous work, the non-activated deposition shows better corrosion resistance because pure nickel starts to form the passive layers at higher corrosion density which is about 20 mA/cm^2 [4]. This is due to the presence of protective layer of Ni-Co-P alloys.

Conclusions

From this study, it can be concluded that anode made by electroless deposition of Ni-Co-P alloy on iron substrate in alkaline bath solution could perform good coating layer without activation and sensitization of the substrates. The coating performance have potential of substituting platinized anodes and Dimentional Stable Anode (DSA)-type electrodes in impressed current cathodic protection system in order to produce more economical system with lower anodic consumption rate if the coating adhesion could be improved. The possible method to obtain that purpose is heat treatment on electroless coated substrates.

References

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