

DEVELOPMENT OF NOVEL CORROSION RESISTANT ELECTROLESS Ni-P COMPOSITE COATINGS FOR PIPELINE STEEL

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ABSTRACT

This paper describes the deposition of various Ni-P composite coatings over an AISI 1012 steel sample through an electroless coating process. The composite coatings were prepared using various ternary additives namely carbon nanotubes, titanium, and alumina. Coated samples were characterized over alongside surface and cross-sections using energy dispersive spectroscopy. Corrosion behaviors of composite coatings were characterized using potentiodynamic polarization. Mechanical and tribological attributes were evaluated using Vickers hardness and nano-indentation, respectively. Among the candidate additives, Titanium reached the maximum incorporation (upto 30 wt.%). Alumina particles showed competing compromise between surface smoothness and deposition rate. Carbon nanotubes improved lubrication effects by reducing co-efficient of friction (checked using universal micro tribometer). Alumina manifested the highest hardness and the least corrosion rate in comparison to the candidate additives.

Keywords: Electroless nickel, Alumina, Carbon nano-tubes, Titanium, Composite coating.

INTRODUCTION

Corrosion and wear resistance has always been among the highly important parameters for equipment and piping in oil and gas facilities.¹⁻² The corrosion is considered as the deterioration of materials as a result of electrochemical reaction with surrounding environment or contained service.³ Wear is defined as the removal and deformation of material due to mechanical interaction between two or more objects.⁴ Increased demand for surface protection and reduced operative costs touts for protective coatings with improved mechanical, electrochemical, and tribological properties.

Electroless nickel phosphorous coatings are known for decades for their wear and corrosion resistance but there is still room to improve their attributes to expand their industrial applications.⁵⁻⁶ Various additives namely ceramics, diamond, graphene, chromium are reported to be co-deposited with Ni-P coatings to achieve ternary Ni-P coatings with improved mechanical and electrochemical properties.⁷⁻⁸ This co-deposition process of electroless coatings does not only rely on the type of the ternary additive, but also on other factors such as size of particle, concentration in the plating bath as well as compatibility of the additive within the coating matrix etc.⁹ Previous studies by Rana et. al. has manifested the improvement in corrosion, mechanical and tribological attributes via incorporation of graphene in the coating matrix.¹⁰⁻¹² On the other hand, graphene's inability towards large scale synthesis impedes its application for industrial facilities.

This research work aims at improving the abovementioned properties of Ni-P coatings via incorporation of various commercially available additives namely Alumina, Titanium and Carbon nanotubes. Carbon nanotubes (CNTs) is considered as a promising material due to its electrical and physical properties such as, thermal, and electric conductivity, tensile strength, and elastic modulus.¹³⁻¹⁴ Alumina on the other hand, is a widely known for its hardness and wear resistance.¹⁵ Whereas Titanium has excellent corrosion behaviors which makes it a suitable choice under erosive-corrosive conditions e.g., in heat exchanger tubes carrying high velocity streams.¹² Moreover, Titanium composites such as NiTi alloys are known for their shape memory, super-elastic properties, as well as wear resistance.¹⁶⁻¹⁷ Various Ni-P composite coatings were synthesized in electroless plating baths via addition of candidate additives followed by the investigation of various parameters and governing mechanisms.

EXPERIMENTAL DETAILS

AISI1012 discs (chemical compositions are mentioned in table 1) were used as a substrate. The disc-shaped (Diameter: 15mm, thickness: 6 mm) coupons was grinded in steps using SiC abrasive papers of various grit sizes namely 240, 320, 400, and 600. The grinded coupons were then polished diamond solution to achieve mirror-like surface finish up to 1 μm . The polished coupons were cleaned with de-ionized water followed by drying and storage in a desiccator.¹⁰

Table 1: Chemical composition of AISI 1012 in wt. %.

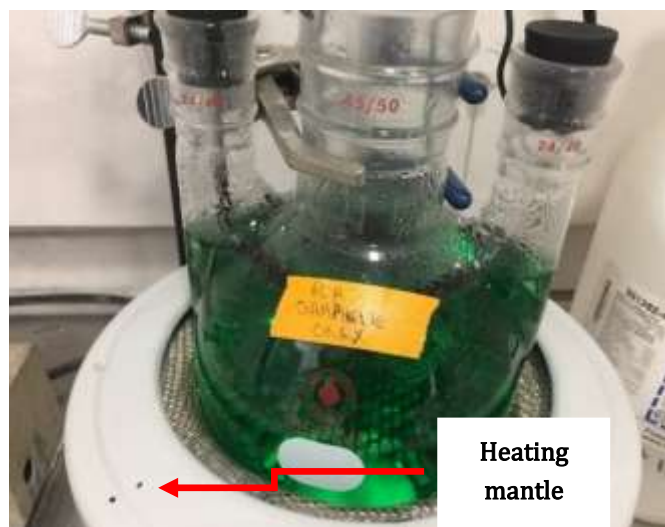
Manganese, Mn	Carbon, C	Sulfur, S	Phosphorous, P	Iron, Fe
0.75	0.175	0.05 max	0.04 max	Balance

The samples were dipped in an Alkaline medium (sodium phosphate 30 g/L, sodium carbonate 30 g/L, sodium hydroxide 50 g/L) at 80°C for 5 minutes (using hot plate and constant monitoring via thermometer) to remove any grease, debris from the surface, followed by rinsing with de-ionized water. After rinsing, the samples were etched via dipping in 1.7 M sulfuric acid for 10 seconds to activate the surface. After etching, the coupons were rinsed again with de-ionized water followed by submerging in electroless plating bath.

The coatings were prepared in two steps namely pre-coating where the samples were dipped in Ni-P plating bath (comprised of deionized water, nickel sulfate as a salt and sodium hypophosphite as a reducing agent) for 30 minutes followed by submerging in a second plating bath containing ternary additive in Ni-P solution. The coating in the second bath were carried out for a duration of 90 minutes. During entirety of plating process, temperature of both plating baths was kept at 89°C. Table 2 summarizes the plating parameters whereas the arrangement for electroless plating is shown in Figure 1.

Table 2 Experimental conditions for electroless plating ⁹:

pH	Temperature	Carbon nanotubes	Alumina	Titanium
4.6 + or - 0.1	89 + or – 1 °C	0.02g/l	7g/l	5g/l

**Figure 1: Electroless plating bath arrangement for Ni-P-Al₂O₃**

After completion of coatings, the coupons were taken of the plating followed by various characterization. The coated coupons were sliced into cross-sections using high precision CBN blade followed by mounting in resin. The mounted coupons were then polished to 1 µm finish followed by EDS analysis to account for chemical composition and deposition thickness.

The cross-sections were also characterized using Vickers Indentation to account for hardness. The coated surfaces were gently polished to Grit 600 finish followed by diamond polishing to eliminate the rough patterns and allow the smooth surface for surface and electrochemical characterizations.

CHARACTERIZATION WORKS

Each sample exposed to 3.5 wt. % NaCl solution (simulating sea water conditions) in a test cell to account for electrochemical behavior. Test cell employed three electrode configurations namely WE (working electrode) which was connected to test specimen, SCE (saturated calomel electrode) as RE (reference electrode) and Pt as CE (counter electrode). OCP (open circuit potential) was conducted for 90 minutes followed by potentiodynamic polarization +/- 1.5 V above and below OCP. From the slope of Tafel plots, corrosion current density was calculated, followed by the determination of corrosion rate(s) via below equation: ¹¹

$$CR = \frac{3.27 \times 10^{-3} \times icorr \times EW}{D}$$

icorr (corrosion current density): µA/cm²

D (density) = 7.87 g/cm³ (AISI standards)

EW (Equivalent Weight) = 27.71 (unitless)

Dry wear testing is used for characterization of various attributes namely thickness, porosity, adhesion, strength, hardness, ductility, stress, and wear resistance. One of the simplest ways for measuring wear from the weight loss during a test. During the test, a tungsten carbide ball with a diameter of 5.3 mm was used as a counter-face material. The ball slides against a flat pre-weighed specimen in a linear, back and forth motion. The ball is mounted inside a ball holder which is attached to a load sensor for controlling and recording forces. Test parameters namely tangential load (Fx), normal load (Fz), wear track depth (Z) were continuously monitored during the test. Different loads were applied between 4N and 10N to all the samples to compare the weight loss and the depth of the tracks. The frequency applied was 4Hz and the time was 10 minutes. The test specimen was weighed with an electronic balance before and after wear test in order to account for weight loss.