

Mechanical Wear and Corrosion Characterisation of Electroless Nickel – Phosphorus Duplex Coating

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Abstract:- In Electroless nickel phosphorus coatings, the corrosion and wear resistance are determined by the percentage weight of phosphorus in the coating. In order to improve the sustainability of Nickel- Phosphorus coating on a substrate subjected to wear and corrosion during use, by using varying temperature and pH levels, bilayer and duplex coating are coated on the sample and a comparative study was conducted along with the conventional mono-layer coating and the results were analyzed. Bilayer and duplex coatings constituted of two layers of 10 micro meters, each were prepared according to three distinct configurations (medium Phosphorous, High Phosphorous and a Duplex layer). The hardness characteristics of duplex coatings are influenced by hardness of Ni₃P phase in the coatings which is found to be more stable and is obtained by heat treatment after the coating process is completed. The duplex coated sample is heat-treated to check whether there is any change in the corrosion properties while increasing the surface hardness. The wear test was conducted on a pin on disc apparatus, corrosion testing method used was salt spray corrosion testing, surface hardness / micro hardness was calculated using a micro Vicker's hardness tester.

Keyword: Electroless nickel – phosphorus, duplex coating, corrosion test, wear test, hardness test.

1. INTRODUCTION

Electroless Nickel plating is more superior to Electrolytic plating because of the comparatively lower costs, more uniformity in coating of Nickel and thus preventing thin spots (less densely coated spots), coating of irregular parts with crevices can be achieved with better coating uniformity using Electroless Nickel plating and the low phosphorous- Nickel coating after heat treatment is much harder to conventionally preferred chromium coating. All this contributes to the wider adoption of Electroless Nickel plating in industries that require higher corrosion, hardness and wear resistances like the automotive and aerospace industries.

The term “Electroless plating” describes the methods of depositing metals and alloys by means of electrochemical reactions. However, chemical plating is the more accurate term that can be used to denote the several means of metal deposition without the application of electric current from an external source. In this process, the chemical reaction proceeds continuously on selected surfaces, providing the means to produce uniform coatings with unique properties on a wide variety of substrates. The chemical deposition of a metal from an aqueous solution of a salt of said metal has an electrochemical mechanism, both oxidation and reduction (redox), reactions involving the transfer of electrons between reacting chemical species. The oxidation of a substance is characterized by the loss of electrons, while reduction is distinguished by a gain of electrons. Further, oxidation describes an anodic process, whereas reduction indicates a cathodic action.

The substrate chosen for coating is EN353 because of its low carbon composition and large industrial application. The

reducing agent used is sodium hypophosphite (NaPO₂H₂•H₂O) which reacts with the metal ions to deposit metal. In general, Electroless Nickel plating is characterized by the selective reduction of metal ions only at the surface of a catalytic substrate immersed into an aqueous solution of said metal ions, with continued deposition on the substrate through the catalytic action of the deposit itself. Since the deposit catalyzes the reduction reaction, the term “autocatalytic” is also used to describe the plating process.

CONSTITUENTS OF ELECTROLESS NICKEL PLATING

A source of nickel ions, a reducing agent, suitable complexing agents, stabilizers/inhibitors, energy The source of nickel ions is “Nickel Chloride Hexahydrate” (NiCl₂•6H₂O). The Chloride anion can act deleteriously when the EN plating bath is used to plate Aluminum, or when the EN deposit is used as a protective coating over ferrous alloys in corrosion applications. The nickel concentration of commercial acid-type (pH 4to 6) Electroless nickel solutions lies within the range of 4.5 to 11 g/L (0.08 to 0.19M). The nickel concentration of EN solutions used for most industrial applications is usually 6.5-11 g/L (0.09to0.13M). When the nickel concentration is equal to or greater than approximately 5g/L (0.085M), it has little or no effect on the plating rate the plating reaction is said to be zero order with respect to the nickel ion concentration. The phosphorus content of Ni-P deposits is influenced by the nickel concentration in the plating baths only when the nickel concentration is less than about 0.1M. If the nickel concentration is increased beyond 0.1M (5.8 g/L), the phosphorus content will remain invariant,

provided that the hypophosphite concentration is held constant.

The reducing agent used is Sodium hypophosphite ($\text{NaH}_2\text{P}_2\text{O}_4 \cdot \text{H}_2\text{O}$). The reducing agent contains two or more reactive hydrogen's and nickel reduction is resulted from the catalytic dehydrogenation of the reducing agent. The reduction of nickel is always accompanied by the evolution of hydrogen gas. Hydrogen ions are generated as a by-product of the reduction reaction. The utilization of the reducing agent for the depositing metal is considerably less than 100 percent. The molar ratio of Nickel deposited to the reducing agent consumed is equal to or less than 1.

The complexing agent used is Ethylene diamine ($\text{H}_2\text{NCH}_2\text{CH}_2\text{NH}_2$). There are three principal functions that complexing agents perform in the EN plating bath:

- They exert a buffering action that prevents the pH of the solution from decreasing too fast.
- They prevent the precipitation of nickel salts, e.g., basic salts or phosphites.
- They reduce the concentration of free nickel ions.

In addition to these functions, complexing agents also affect the deposition reaction and hence the resultant nickel deposit. The chemical properties of nickel ions in aqueous solution are altered when they are combined with complexing agents. Some of the common properties of solvated nickel ions that can be affected are color, reduction potential, and solubility. The effects complexing agents have on some of the common properties of nickel ions, such as color, reduction potential and solubility.

An Electroless nickel plating solution can be operated under normal operating conditions over extended periods without adding stabilizers however; it may decompose spontaneously at any time. Bath decomposition is usually preceded by an increase in the volume of hydrogen gas evolved and the appearance of a finely-divided black precipitate throughout the bulk of the solution. This precipitate consists of nickel particles and nickel phosphide. The metal ions will accumulate in any production facility until their concentration exceeds their respective metal hydroxide solubility product limits and begin to precipitate.

Catalytic reactions, such as Electroless nickel plating, require energy to proceed. The energy is supplied in the form of heat. Temperature is a measure of the energy (heat) content of the plating solution. The coating process was carried out at 90°C. Before performing Electroless nickel plating, the material to be plated must be cleaned by a series of chemicals; this is known as the pre-treatment process. Failure to remove unwanted "soils" from the substrates surface results in poor plating. Each pre-treatment chemical is followed by water

rinsing (two to three times) to remove chemicals that may adhere to the surface. De-greasing removes oils from the surface, whereas acid cleaning removes scaling.

Activation is done with a weak acid etch, or nickel strike or, in the case of non-metallic substrate, a proprietary solution. After the plating process, plated materials must be finished with an anti-oxidation chemical like disodium phosphate, followed by water rinsing to prevent staining. The rinsed object must then be completely dried or baked to obtain the full hardness of the plating film.

The heat treatment process of the coated substrates is carried out to further improve the coatings hardness and wear properties. Heat treatment allows the changing the mechanical properties of the substrate without altering its original form. The heat treatment process is done in a furnace at 400°C temperature while passing air.

II. EXPERIMENTAL AND TESTING METHODOLOGY

The process of Electroless nickel plating to a metal substrate is an autocatalytic chemical reduction, which means that instead of using an outside source of electricity like in the conventional Electroplating process, the Electroless Nickel plating process uses a chemical bath to deposit a Nickel / phosphorous layer onto the metallic surface. A surface coated in Electroless nickel can even be used on non-conductive surfaces which allows for plating of a wider variety of base materials. This Electroless process greatly improves the objects resistance to mechanical wear and leaves a more uniform nickel coating for high-precision and low tolerance parts, which can be applied to both ferrous and non-ferrous substrates of any geometry or intricate shapes.

The tank material used is the natural, stress-relieved polypropylene (PP), as it is inert (non-reactive) to the plating solutions generally used. Tanks can be easily fabricated and when using proper nitrogen welding technique, they are fairly reliable. Polypropylene is also used in special plumbing or attached fixtures. The relative low cost and versatility of design considerations make polypropylene the most widely used material of construction for Electroless nickel plating purpose tanks. Polypropylene is not without its disadvantages though. These tanks have a finite life span, which can be approximated by taking the inverse of the tank wall thickness (e.g., 1/2-in. liners usually last 4 to 8 years, while 3/4-in. tank walls will need to be replaced in roughly half that time). The size of the tank also influence its useful life. Generally, larger tanks have shorter lives. Polypropylene Electroless nickel tanks should be enclosed in outside support tanks for safety. As the support tank must be substantially larger, to accommodate the polypropylene expansion (because of heating during plating process), the liner corners are

subjected to extreme pressures both while cold and during heat-up and operation. Polypropylene-lined Electroless nickel tanks have been fabricated in sizes up to 20 feet long and 10 feet wide.

Liners are used to protect the tanks from etching and corrosion due to prolonged exposure to chemicals during plating. The liner used here is Polyvinylchloride (PVC). They are preferred over coated tanks for being a cheaper alternative, easy installation with minimal down time and the loss of production. It is also custom made to tank dimensions for a proper fit and the elimination of sandblasting and bonding of liner to tanks. Liners will expand and contract with temperature changes and tank movement. The material (Polyvinylchloride) is thin and lightweight and designed to be used as a disposable method of lining the tank. It eliminates the need to clean the tank with Nitric Acid after use.

Stainless steel IS5522 is usually used for electric immersion heaters. Heaters are used for maintaining the solution at a optimum temperature of around 90oC. The main advantages of immersion heaters are a low front-end capital cost and ease of installation. The major disadvantages are potential plate out on the heaters, as well as a potential fire hazard when the solution level drops too low. Automatic controls should never be installed on electric immersion heaters for safety purposes. Electrical shocks and stray current plating problems will be caused by improperly grounded heaters or resistance coils to stainless steel sheaths, causing electrical shorting.

Filtration of Electroless nickel baths should be continuous and at a rate of at least 10 times per hour. A 1 µm filter is adequate. It should be noted that because of the geometry of a rectangular tank, a filtration rate (tank turnover) of 100 times/hr would not expose all the solution to the filter. Filters should be changed daily. There are a number of filter systems available, which can be divided into two general groups as free flowing filters and closed chamber filters. The free-flowing filter used in our setup is a bag-type polypropylene felt bag. These filters are usually the most cost effective. Filter chambers for bag filters is used here. These provide better use of tank space; however, the advantage of being able to inspect the filter and observe what is going on in the bath is lost since it's placed outside the bath.

Most Electroless nickel pumping systems are currently either Chlorinated polyvinyl chloride (CPVC) or stainless steel. CPVC has been used in our setup. CPVC has proven itself to be an adequate material in most low volume/low pressure applications-a category that includes most of the Electroless nickel baths. A good limit for CPVC impellers is 200L/min. Pump bodies may still be made of easily molded CPVC. When flow rates surpass 400 L/min (1 00 gal/min), pressure becomes a definite factor. Few plastics can give adequate,

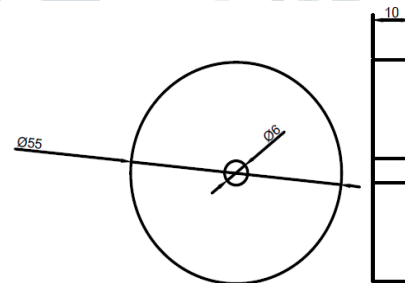
cost effective service under these conditions and therefore stainless steel must be used. There are many options as to what type of stainless steel should be required. No matter what type of stainless steel is used, it will be subjected to plating. Therefore, periodic passivation with nitric acid is required, preferably every day.

III. EXPERIMENTAL SAMPLE AND COATING COMPOSITION:

The metallic substrate used in the coating process is the low carbon content EN353 steel with wide applications where mechanical properties like wear, corrosion and abrasion resistance are much desirable properties. This grade is in general used for a lot of automobiles functions like heavy duty tools, shaft, pinion, cam shafts and gudgeon pins. This EN353 alloy Steel is used as a connector between the pipes at the middle. They are also used as a plug in to resist pressure in Pressure Valves. They have a huge demand in many large scale industrial purposes where superior wear and corrosion resistant properties are desirable.

EN353 Composition

C	Si	Mn	Ni	Mo
0.18	0.23	0.65	1.20	0.45



Sample dimensions

COATING COMPOSITION:

SAMP LE NO	COATING DESCRIPTI ON	COMPOSITION	HEAT-TREA TMEN T
1	Monolayer Ni-P	Ni - 89.4% P - 10.6%	No
2	Bilayer Ni-P	Layer 1: Ni - 89.4%, P- 10.6% Layer 2: Ni - 89.4%, P- 10.6%	No
3	Bilayer Ni-P	Layer 1: Ni - 89.4%, P- 10.6%	Yes

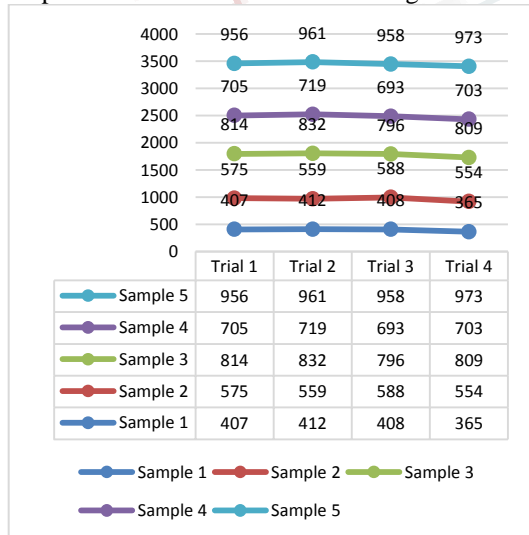
		Layer 2: Ni - 89.4%, P- 10.6%	
4	Duplex Ni-P	Layer 1: Ni - 89.4%, P- 10.6% Layer 2: Ni - 86%, P- 14%	No
5	Duplex Ni-P	Layer 1: Ni - 89.4%, P- 10.6% Layer 2: Ni - 86%, P- 14%	Yes

IV. RESULTS AND DISCUSSION

Various tests have been done to determine the coatings mechanical wear, corrosion and other properties to draw to the conclusion of whether the expensive duplex coating justifies its use over the conventionally opted for mono-layer coating in the industries. The results of each test are discussed in detail and extensively to conclude on the result of the experiment.

VICKERS MICRO HARDNESS TEST:

The Vickers Hardness of the samples was measured using Wilson Wolpert Vickers Hardness tester. For every sample four readings were taken by applying a load value of HV0.025 which equals to about 2N force. It is also observed that sample 3 has higher mean hardness value than sample 4, proving that heat treatment yields the more stable Ni3P phase which shows a higher hardness value. Sample 2 has a higher mean hardness value than sample 1 which shows that duplex coating results in higher hardness over mono-layer coating, despite both the samples being coated with the same medium-phosphorous layer known for wear resistance. A Graph is drawn to illustrate the readings obtained in the trials.



From the graph, sample 5 has the highest hardness when compared to all other samples.

HIGH-TEMPERATURE PIN ON DISC WEAR TEST:

The mechanical wear resistance properties of the samples are compared to determine any increase in the wear properties of the duplex coated samples. A duplex coated and hardened sample must yield higher wear resistance and lower wear volume as compared to the non-hardened and mono-layer coated sample.

SAMPLE NO.	WEIGHT(g)	
	Before testing	After testing
1	149.40	149.35
2	148.90	148.87
3	148.90	148.88
4	153.95	153.44
5	146.11	146.10

FORMULAS:

Wear Volume= Weight loss/Density

Wear Rate=Wear volume/Sliding distance

Sliding distance=circumference x time

$$= 2\pi RN/60 \times \text{Time}$$

Wear resistance=1/Wear ratio

Density of substrate (EN353) = 8.08 g/cm³

CALCULATIONS:

Sliding distance= $2\pi \times 2.75 \times 1000/60 \times 5$

$$= 1439.9 \text{ cm/min}$$

SAMPLE 1:

Wear volume= $149.40-149.35/8.08=6.19 \times 10^{-3} \text{ cm}^3$

Wear rate= $6.19 \times 10^{-3}/1439.9=4.3 \times 10^{-6} \text{ cm}^2$

Wear resistance= $1/4.3 \times 10^{-6}=232687.84 \text{ cm}^{-2}$

SAMPLE 2:

Wear volume= $148.9-148.87/8.08=3.7 \times 10^{-3} \text{ cm}^3$

Wear rate= $3.7 \times 10^{-3}/1439.9=2.57 \times 10^{-6} \text{ cm}^2$

Wear resistance= $1/2.57 \times 10^{-6}=389162.2 \text{ cm}^{-2}$

SAMPLE 3:

Wear volume= $148.9-148.88/8.08=2.48 \times 10^{-3} \text{ cm}^3$

Wear rate= $7.43 \times 10^{-2}/1439.9=1.72 \times 10^{-6} \text{ cm}^2$

Wear resistance= $1/1.72 \times 10^{-6}=581719.6 \text{ cm}^{-2}$

SAMPLE 4:

Wear volume= $153.95-153.93/8.08=2.5 \times 10^{-3} \text{ cm}^3$

Wear rate= $2.5 \times 10^{-3}/1439.9=1072 \times 10^{-6} \text{ cm}^2$

Wear resistance= $1/1.72 \times 10^{-6}=581719.6 \text{ cm}^{-2}$

SAMPLE 5:

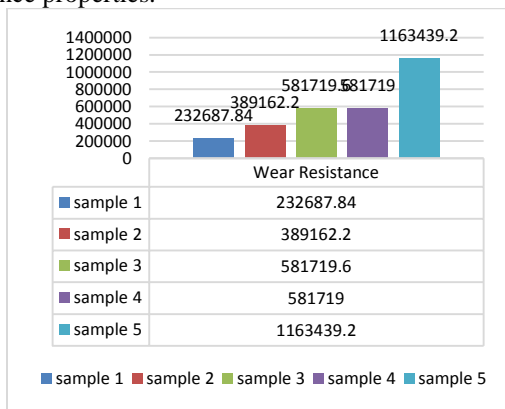
Wear volume= $146.11-146.10/8.08=1.24 \times 10^{-3} \text{ cm}^3$

Wear rate = $1.24 \times 10^{-3} / 1439.9 = 0.86 \times 10^{-6} \text{ cm}^2$
 Wear resistance = $1 / 0.86 \times 10^{-6} = 1163439.2 \text{ cm}^{-2}$

Maximum wear resistance has been clearly exhibited in sample 5 over the rest of the samples which implies the superior wear resistance properties of Duplex coated substrate over the single coated sample.

WEAR RESISTANCE:

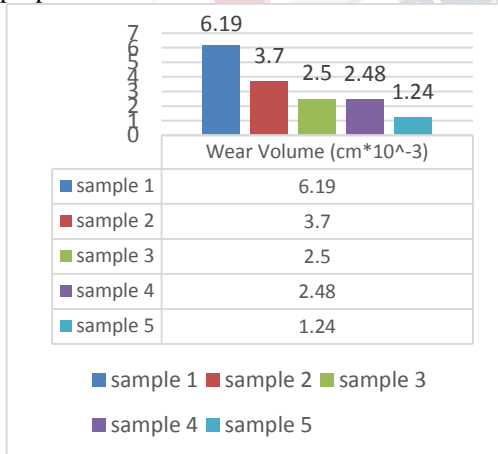
Wear resistance is the inverse of Wear ratio or simply defined as the resistance to mechanical wear. A higher wear resistance value indicates superior wear resistance properties.



Sample 5 has shown significant wear resistance over its counterparts indicating optimum wear resistance properties in high-wear applications.

WEAR VOLUME:

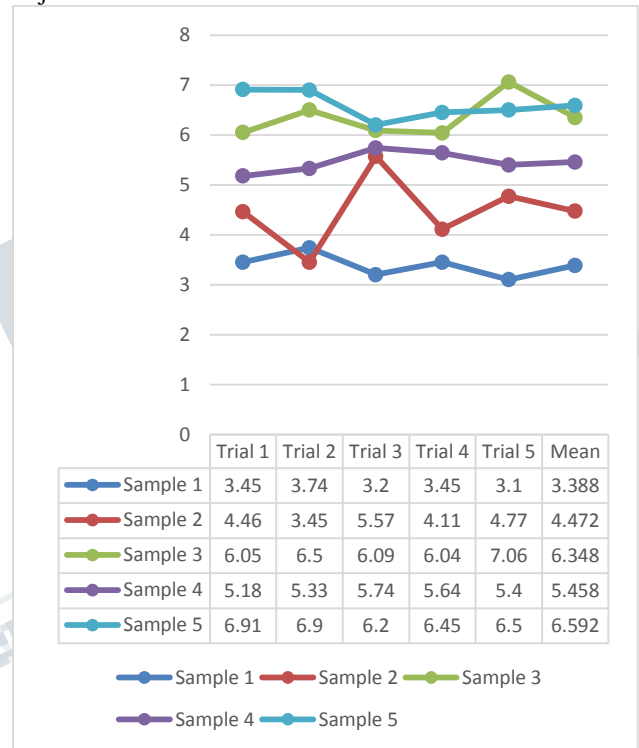
Wear volume is defined as the ratio of weight loss to density of the substrate. The wear volume indicates the material loss calculated in terms of volume. The material with the least wear volume loss is of highest wear resistance properties.



The above graph shows the least wear volume of sample 5 indicating towards its superior wear resistance properties.

FRICTIONAL FORCE:

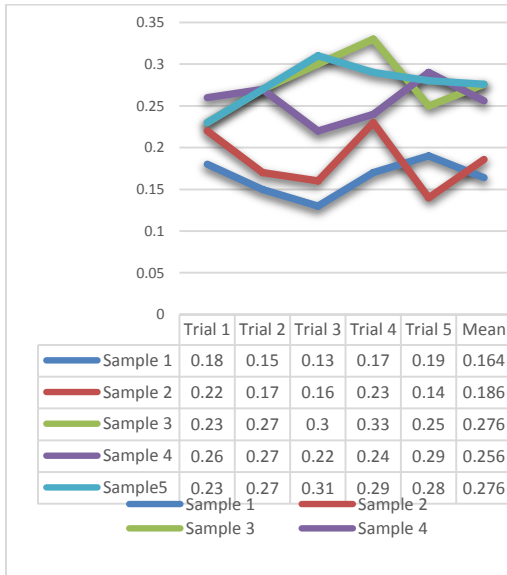
Frictional force refers to the force generated by two surfaces that contacts and slide against each other. These forces are mainly affected by the surface texture and the amount of force impelling them together. The angle and position of the object affect the amount of frictional force.



In this experiment, we have considered dry friction. Dry friction is the friction between any two objects that are in mechanical contact and is normally assumed to be the influence of dry friction force. This force is generated in the tangential direction of the plane of contact, and both prevent the movements between the surfaces

CO-EFFICIENT OF FRICTION:

The co-efficient of static friction is the minimum force required to get an object to slide on a surface, divided by the force pressing them together. There are no units associated with coefficient of friction.



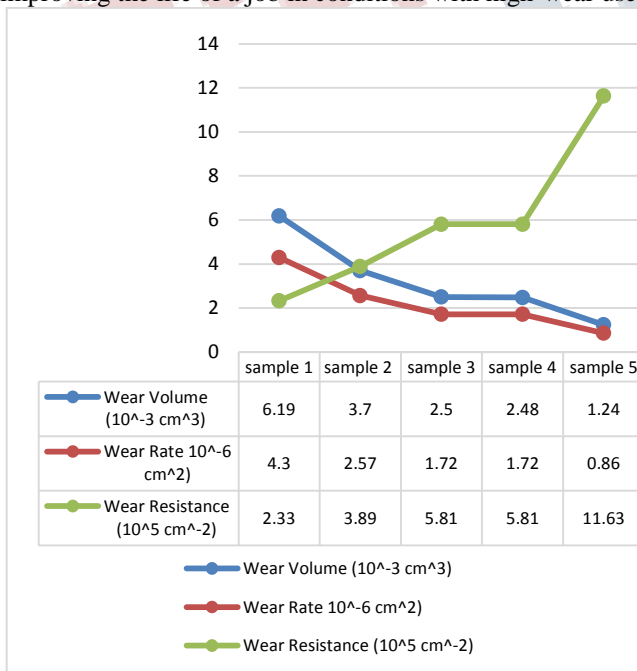
SALT SPRAY CORROSION TEST:

The salt spray test results indicate the coating's resistance to corrosion in the real world. Though there exists no direct correlation between the test hours and real-time applications, a significant increase in corrosion time was observed in the duplex coating as compared to the mono layer coating.

Sl.NO.	PARAMETERS	SPECIFICATIONS	ACTUAL RESULTS
1	Concentration of the solution	5-6%	5%
2	Specific Gravity	1.0255 to 1.040	1.028to1.029
3	Quantity of fog collected	1 to 2 ml/hour	1.3 ml/hour
4	pH of collected fog	6.5 to 7.2	6.97to 7.00
5	Cabinet Temperature	33 to 37 °C	34.8 to 35 °C
6	Air Pressure	12.0 to 18.0 psi	15 psi
7	Titre Value	3.4 to 5.1 ml	4.0 ml

WEAR PROPERTIES OF THE SAMPLES:

The graph summarizes on the different properties calculated to determine the sample with the most superior wear resistance properties. From the graph, we can conclude that Sample 5 shows the most superior wear resistance properties and that using a duplex coating can significantly improve the wear properties of the substrate thereby decreasing its susceptibility to wear and abrasion and significantly improving the life of a job in conditions with high-wear use.



SAMPLE 1(PRE-TESTING):



Sample 1 before Testing

SAMPLE 1(POST-TESTING):

After 24 hours of Testing.



Sample 1 after 24 Hours

SAMPLE 2 (PRE-TESTING):



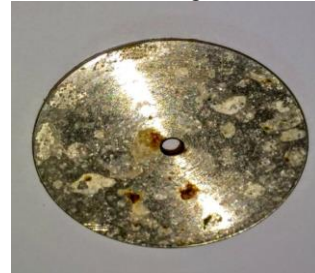
Sample 2 before Testing

SAMPLE 2 (POST-TESTING):
After 24 hours of Testing



Sample 2 after 24 Hours

SAMPLE 4 (POST-TESTING):
After 72 hours of Testing.



Sample 4 after 72 Hours

SAMPLE 3 (PRE-TESTING):



Sample 3 before Testing

SAMPLE 5 (BEFORE TESTING):



Sample 5 before Testing

SAMPLE 3 (POST-TESTING):
After 72 hours of Testing.



Sample 3 after 72 Hours

SAMPLE 5 (POST-TESTING):
After hours of Testing.



Sample 5 after 72 Hours

SAMPLE 4 (PRE-TESTING):



Sample 4 before Testing

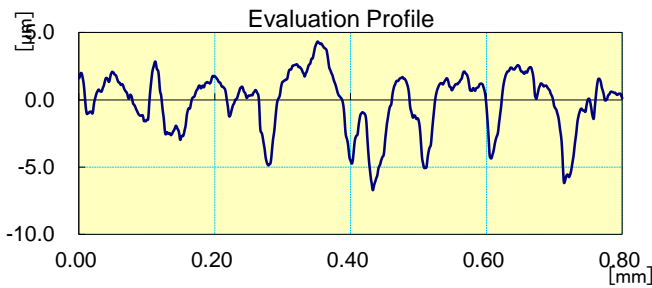
From the above images, we infer that the high-Phosphorous duplex coating on Sample 4 shows maximum corrosion resistance with the sample being corroded beyond use after only 72 hours and the first spot of rust being visible at 60 hours of testing time while for sample 5 the first rust spot was observed at 60 hours and few more spots started showing up at 96 hours. Sample 2 also duplex coated with medium phosphorous shows significantly better corrosion resistance over the conventionally used mono-layer of Electroless Nickel coating on Sample 1, with the sample being corroded at 16 hours of testing time as compared to the 12 hours of Sample 1.

The test results clearly show the superior corrosion resistance properties being exhibited in the Samples coated with the Duplex layers over mono-layer coating under the same testing conditions.

ROUGHNESS TESTS:

Roughness is an important parameter when trying to find out whether a surface is suitable for a certain purpose like wear resistance. The following results are obtained when the samples are tested for wear.

SAMPLE 1 PROFILE:

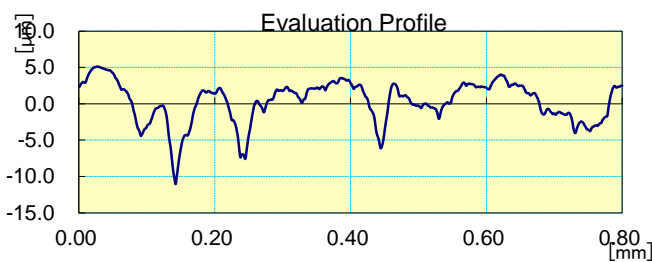


Roughness profile of Sample 1

Ra	1.735
Rq	2.235
Rz	11.031

Roughness Data of Sample 1

SAMPLE 2 PROFILE:

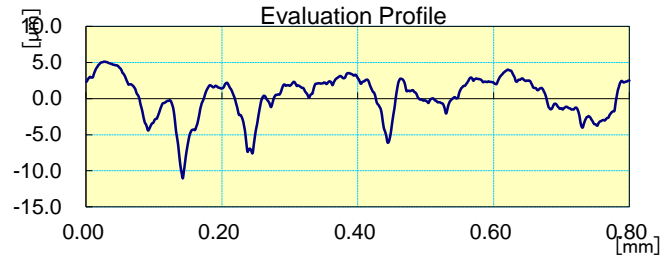


Roughness profile of Sample 2

Ra	2.325
Rq	2.876
Rz	16.150

Roughness Data of Sample 2

SAMPLE 3 PROFILE:

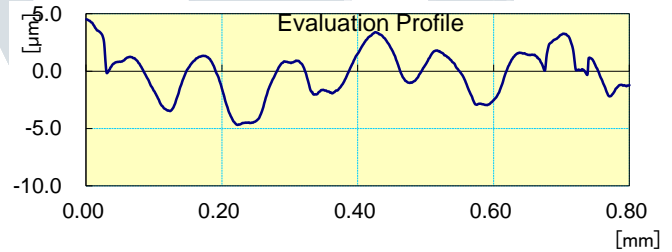


Roughness profile of Sample 3

Ra	2.015
Rq	2.576
Rz	15.950

Roughness Data of Sample 3

SAMPLE 4 PROFILE:

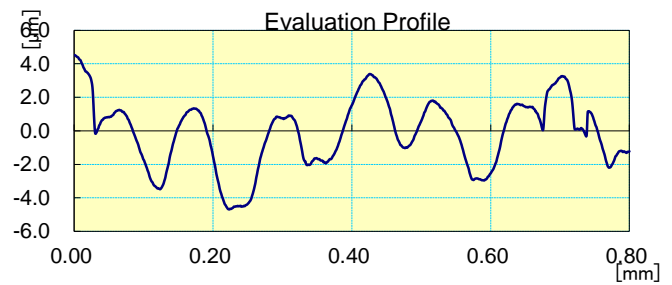


Roughness profile of Sample 4

Ra	1.685
Rq	2.072
Rz	9.211

Roughness Data of Sample 4

SAMPLE 5 PROFILE:



Roughness profile of Sample 5

Ra	1.655
Rq	2.043
Rz	9.084

Roughness Data of Sample 5

The surface roughness test of the substrate coated by Electroless Nickel was done to correlate a rough surface with the wear and corrosion properties. A rougher surface has an inverse effect on the wear properties and is characterized by cracks and fissures which promote corrosion at these points decreasing the overall corrosion resistance of the surface coating.

The mean roughness values (Ra) of all the samples are invariably equal which means the wear and corrosion properties of the results can be compared without any discrepancy in any one of the coated samples.

V CONCLUSION

In this experimental study, we planned to attain both wear and corrosion resistant property in a single coating by employing a coating consists of two layers of different composition. Both the layers have different in their composition i.e. the weight of nickel and phosphorus is different. This coating of two different layers is known as duplex coating. We have used EN 353 as the base metal to be coated and the coated metal was tested for its properties through various equipment and was compared with that of a standard coating. We obtained the following results,

1. **Vickers Hardness:** The Vickers Hardness Number for the samples were calculated by applying a load of HV0.025. From the values obtained it is clear that sample 5 has the highest hardness and it is twice as hard as the normal NiP coated sample 1. Hence it proves that the heat treated duplex coating has superior hardness than normal coating.
2. **Wear Test:** The wear characteristics of the samples were tested on the pin on disc and the results are as follows
 - 2.1. **Wear Volume:** Sample 5 had the least Wear volume when compared to all other samples. So, heat treated duplex coating is better than normal coating.
 - 2.2. **Wear Rate:** Wear rate was found to be gradually decreasing from sample 1 to sample 5. So, sample 5 has the least wear rate when compared to all other samples.
 - 2.3. **Wear Resistance:** The value of wear resistance was observed to be increasing from sample 1 to sample 5. So,

sample 5 has the highest resistance to wear when compared to all other samples

2.4. **Wear:** From the above results, it is clear that sample 5 is highly resistant to wear than all other samples. Hence Heat treated duplex Coating is better in wear resistance when compared to normal NiP coating.

3. **Corrosion Resistance:** The salt spray test showed that the conventional coatings were resistant to corrosion for 16 hours while the duplex coating was resistant till 60 hours and the heat-treated Sample 5 was resistant up to 72 hours. So, the duplex coatings were superior in corrosion resistance when compared to conventional coating.

4. **Roughness:** The Average roughness value was found to be the lowest for sample 4 and 5. So Duplex coating was found to smoother than normal coating.

From all these test results, it is proved that Electroless Nickel Phosphorus duplex coating is better in all aspects when compared to conventional coating. The duplex coated sample high corrosive resistance and high surface rigidity.

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