Original Research Article

COMPARATIVE PERFORMANCE OF COATINGS FOR ASTM A106B LOW ALLOY STEEL

ABSTRACT

Thestudy investigatesthe coating performance of oil pipelines. To examine the coating parameters, different type of coatings were developed based on paint group and electroless nickel phosphorous group (Electroless Ni-P) with or without nano additives. The substrate considered waslow alloy steel ASTM A106B oil pipelinesample. The comparative performance of coatings wasreportedand concluded through mechanical, characterization and corrosion resistance evaluation.

The substrates after chemical and mechanical polishing were coated with paint group comprised of organic paint, zinc paint and aluminum oxide paint with or without nano additives like nano Al2O3, nano ZnO and nano CuO. Similarly, Electroless Ni-Pgroup coated samples were also prepared for the same nano additives. To identify the coating performance different concentrationsof nano additives i.e 0.5, 1, 1.5 and 2 % were usedagainst paint and Electroless Ni-P group. Based on the concentration performance, 1 % nano additive was reportedwith optimum performance establishedthrough surface finish, micro hardness and corrosion resistance values.

The characterization evaluation of the coated samples was studied using Scanning Electron Microscope and Optical Microscope to study surface morphology. To confirm the percentage of elements in coatings, Energy Dispersive X-ray test was also performed. To examine corrosion rate, three different test such as Atmospheric exposure test, Potentio-stat test and wet/dry test were also conducted. To evaluate the mechanical evaluation, hardness test, surface roughness test and coating thickness test were conducted.

After investigation, it was revealed that the overall investigation of Electroless Ni-P group was better than the paint group. The observed resultsdemonstrates the mechanical and characterization evaluation for nano Al2O3 better than the other nano additives. However, coating efficiency was around 24.7 % it means rest of nickel was wasted in the electrolyte. To evaluate the corrosion rate , Electroless Ni-P with nano Al2O3 was 0.0353 mmpy followed by Electroless Ni-P with nano ZnO was 0.0412 mmpy and for Electroless Ni-P with nano CuO was 0.0471 mmpy. After adding Electroless Ni-P with nano Al2O3, corrosion rate was improved by 58.12 % as compared to without addition of nano particles.

Key words: nano additives, corrosion, tribology, surface morphology, low alloy steel

1: INTRODUCTION

Corrosion is the oxidation of a metal due to an electrochemical reaction. To protect the pipeline from the corrosion, the very first attempt is to isolate the pipeline surface from surrounding, so that no corrosion could occur. To avoid this phenomena, coating technique application on pipeline surface would be an effective solution. Currently to protect pipelines against corrosion, different layered fusion bonded epoxy (FBE) coatings and a three-layer polyolefin (3LPO) (polyethylene or polypropylene [1]are in use, but one of the disadvantage of these coatings are at high ambient temperature especially in desert region formation of brittleness in coatings are observed , resulted in cracks development causes thermal-oxidative degradation.

Electroless Nickel phosphorous (Electroless Ni-P) coating is one of the emergent coating technique widely used in aerospace, automobile, electrical and chemical industries worldwide with positive results. However, in oil and gas industry not much research work has been done. Combination of nano additives and Electroless Ni-P coating techniques are also not tried out to a greater extent. Benefit of using nano additives in coating is when nano additives are added in the coating they spread and dissolve uniformly on the layers of coatings due to high volumeto surface area of nano additives since the particle sizes that considered will be from 40 to 50 nm. These nano particles does not allow the oxidation to takes place on the protective barrier of coatings. As all the nano particles chosen for investigation are from oxide group (nano CuO, nano Al2O3,nano ZnO or nano SiO2) these oxides form a passive layer on the coating surface, preventing corrosion. Another factor is, the dissolved nano particles reduces the direct exposure of coating area to the environment, resulting lesser reaction on the surface and hence less weight loss. These nano additives will work like solid particles thus reducing high ambient temperature effect on coatings and also improves surface finish and hardness.

In 1844, Wurtz found out that the metallic nickel could be deposited from an aqueous solution of its salt by reduction with hypophosphite [2]. But the metal which was formed was in powder form. Later, researchers Breteau [3] and Roux [4] took bright coatings on all surfaces in a reaction vessel when the reducing agent had been added. However, these baths decomposed spontaneously and formed deposits on any surface that interacted with the solution, even on the walls of the container. Other investigators analyzed the process but their interest revolved around the chemical reaction instead of the plating process. The discovery of electroless nickel technology as it is used today was developed in 1946 by Brenner et al [5]. Brenner was the first to refer the process as ‘Electroless’. Some of the outstanding characteristics of Electroless Ni-P coatings are superior corrosion and wear resistance, excellent uniformity, wide range of thickness, good solder ability and improved mechanical and physical properties [6]. The current trend is mostly focused on improving the tribological as well as mechanical properties of the Electroless Ni-P coatings that covers fundamental aspects like nickel recovery efficiency, addition of reducing agents, changing EN bath composition and techniques through surfactants, heat treatment , special treatments and mechanical properties.The composition of EN-P bath and there components are shown in the Table 1.

Components	Functions
Nickel Ion source of metal	Reducing agent source of electrons
Complexants stabilizes the solution	Accelerators activates reducing agent
Buffers controlling pH (long term)	pH regulators
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Table 1. EN bath composition and their function [7]

In the present investigation,coatings were developed based on EN-P group and paint group that includes Organic paint, Aluminum oxide paint and Zinc paint. These groups were tried with and without nano additives of nano Al2O3, nano CuO and nano ZnO size 40 to 50 nm. Different nano additive concentrations of 0.5,1,1.5 and 2 % in the bath were tried to evaluate the optimum concentration.Despite the complicated behavior of the deposition reactions, qualitative discussions on the effects of adding nano additives werealso focused. Moreover, the coating performance indicators wereinvestigated and reported in this paper gauged against mechanical evaluation, characterization evaluation and corrosion resistance test.

2: EXPERIMENTAL DETAILS

Experimental work was divided into two stages, Stage I for paint group and stage II for EN-P group. 39 samples for both the stages of size 50 x50 x7 mm3 and 20 x20 x7 mm3 were developed from ASTM A106B low alloy steel[8] used in oil pipelines. Samples were prepared for paint and Electroless Ni-P groups as per the following format as shown in table 1 and 2 below.

Stage I - Paint group

Paint Group Coating Notations With nano additives	Coating Type (Paint group with/without nano additives)	Qty / size of samples for Atmospheric Test in mm3	Qty / size of samples for Dry/Wet, Potentiostat Test in mm3
(A-Z)	Aluminum paint with nano ZnO	1(50 x50 x7)	1(20 x20 x7)
(Z - z)	Zinc Paint with nano ZnO	1(50 x50 x7)	1(20 x20 x7)
(O-Z)	Organic Paint with nano ZnO	1(50 x50 x7)	1(20 x20 x7)
(O-C)	Organic Paint with nano CuO	1(50 x50 x7)	1(20 x20 x7)
(A-C)	Aluminum paint with nano CuO	1(50 x50 x7)	1(20 x20 x7)
(Z-C)	Zinc Paint with nano CuO	1(50 x50 x7)	1(20 x20 x7)
(O-A)	Organic Paint with nano Al2O3	1(50 x50 x7)	1(20 x20 x7)
(Z-A)	Zinc Paint with nano Al2O3	1(50 x50 x7)	1(20 x20 x7)
(A-a)	Aluminum Paint with nano Al2O3	1(50 x50 x7)	1(20 x20 x7)

	Without coating	1(50 x50 x7)	1(20 x20 x7)
Without nano additives	Aluminum paint	1(50 x50 x7)	1(20 x20 x7)
	Zinc Paint	1(50 x50 x7)	1(20 x20 x7)
	Organic Paint	1(50 x50 x7)	1(20 x20 x7)
	Without Coating	1(50 x50 x7)	
	Total Qty	14	13

Table 2: Low Alloy steel samples for paint group

Stage II – Electroless Ni-P group

EN-P group Coating Notations	Coating Type (EN-P group)	Qty / size of samples for Atmospheric Test in mm3	Qty / size of samples for Dry/Wet Test in mm3	Qty / size of samples for Potentio-stat Test in mm3
With/without nano additives	EN-P without nano additive	1(20 x20 x7)	1(20 x20 x7)	1(20 x20 x7)
	EN-P with nano ZnO	1(20 x20 x7)	1(20 x20 x7)	1(20 x20 x7)
	EN-P with nano Al2O3	1(20 x20 x7)	1(20 x20 x7)	1(20 x20 x7)
	EN-P with nano CuO	1(20 x20 x7)	1(20 x20 x7)	1(20 x20 x7)
	Total Qty	4	4	4

Table 3: Low Alloy steel samples for EN-P group

2.1 Sample Cleaning

Samples were primary polished with wet 400, 600, 800 and 2000 grit SiC paper. To avoid poor coating and to have excellent adhesion, the surface of the samples were cleaned with distilled water. Later it was cleaned with Acetone followed with rinsing again in the distilled water. Further, ultrasonic cleaning was done for three minutes with Methanol followed washing with distilled water. For sample preparation of EN deposit, sulfuric acid was used with concentration of 8%. Similarly for the paint coating of primer paint, red oxide was used for the sample coated with Aluminum, Zinc and Organic Paint as shown in figure 1 below.

Figure 1: Samples after Mechanical / Chemical polishing and after applying primer

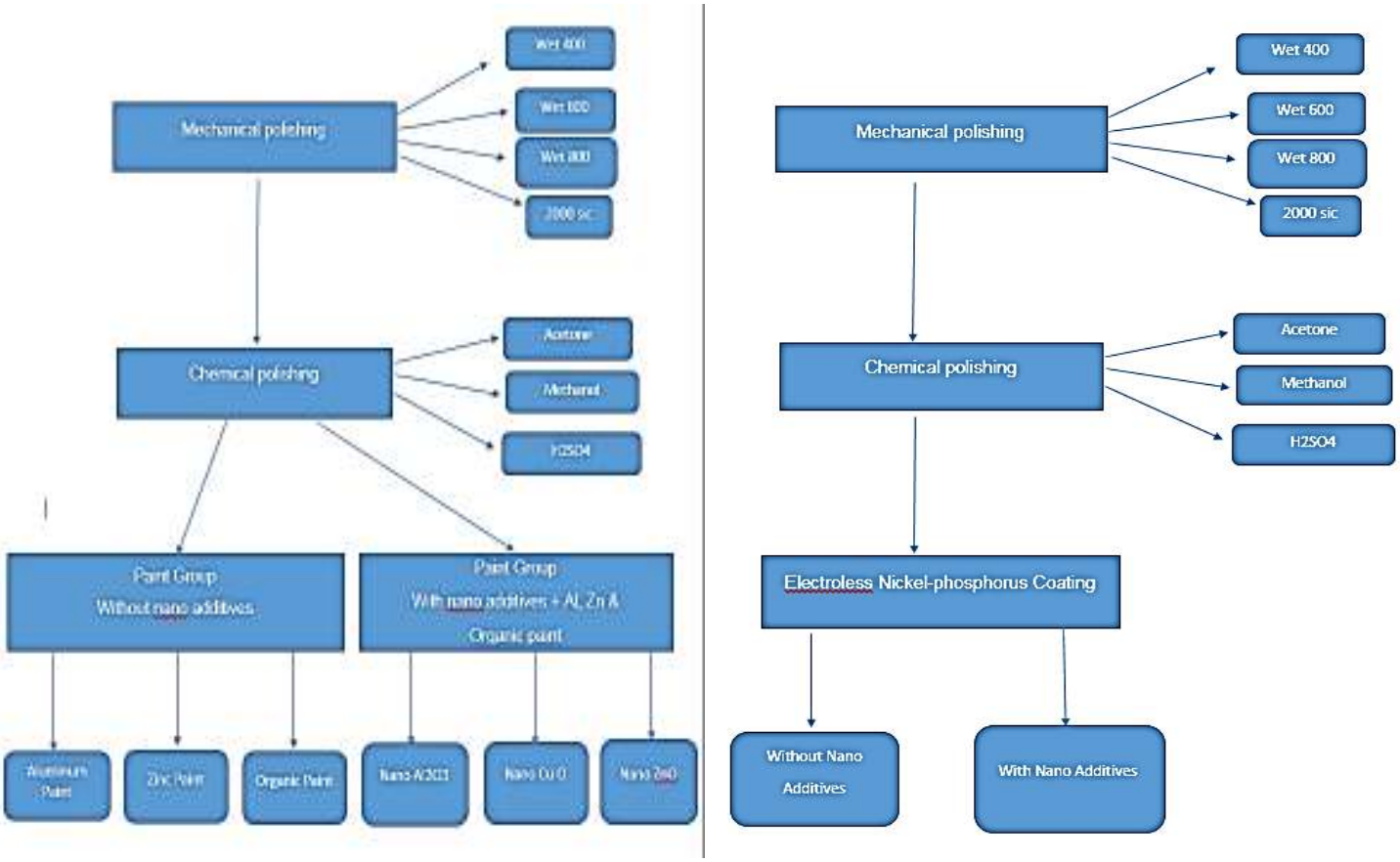


Figure 2: Mechanical and chemical polishing for stage I and II

2.2Coating of Samples

Stage I - Paint group

For primer base, the samples were coated with primer paint (Red Oxide). Samples after mechanical and chemical polishing and after primer organic, zinc and aluminum paint were applied to prepare the samples. Similarly for nano additives samples after polishing and after red oxide before application, 50 ml of each paint was mixed with 0.5 gms of nano additives like ZnO, CuO and AL2O3. Both were mixed and put for stirred for 2 hours, so that proper missing of paints with nano additives could be achieved. Sample configuration is shown in Figure 2,3 and 4.

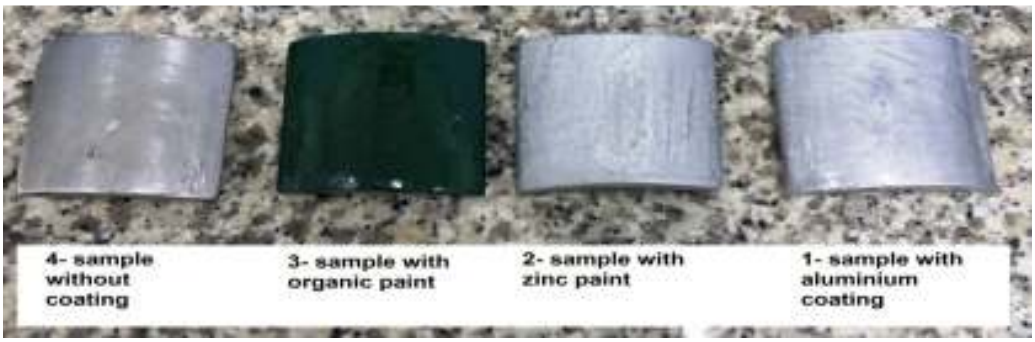


Figure 3: Samples after paint coating

Figure 4: Samples after organic and aluminum paint with nano additive

Stage II – Electroless Ni-P group

The application of EN-P deposited for coating samples were prepared in two baths for stage one and two separately. Bath A was without nano additives and Bath B with different nano additives considering concentration combination of 0.5,1,1.5 and 2 %.Based on the concentration combination, 1 % gives the optimum value. The results reported in the figures and tables refer 1 % optimum concentration value of nano additives. Figure 5 and table 4 below explains the procedure adopted and composition required in preparing the EN-P bath. Similarly figure 6 to 8are the Electroless Ni-P coated samples with and without nano additives.

Particulars	Bath A (200ml)	Bath B (200ml)	Description
Nickel Chloride	(6g/200ml)	6g/200ml	Source of Ni - metal
Sodium hypophosphite	(8g/200ml)	8g/200ml	Reducing Agent
Sodium tri citrate	(5g/200ml)	5g/200ml	Stabiliser – prevent solution breakdown
Ammonium Chloride	(10g/200ml)	10g/200ml	Complexing agent – stabilize the solution
Ammonia Solution	5 ml	6 ml	To maintain the pH value
Palladium Chloride		0.5 g	Striking agent (if required)
Nano Copper oxide <50nm		(0.2 g/200ml)	4 different nano concentrations considered, 0.5,1,1.5 and 2 %. Optimum result found against 1 % concentration for 0.2 gms.
Nano Aluminum oxide <50nm		(0.2 g/200ml)	
Nano Zinc oxide <50nm		(0.2 g/200ml)	

Table 4: Electroless Ni-P solution bath preparation for stage II [9]

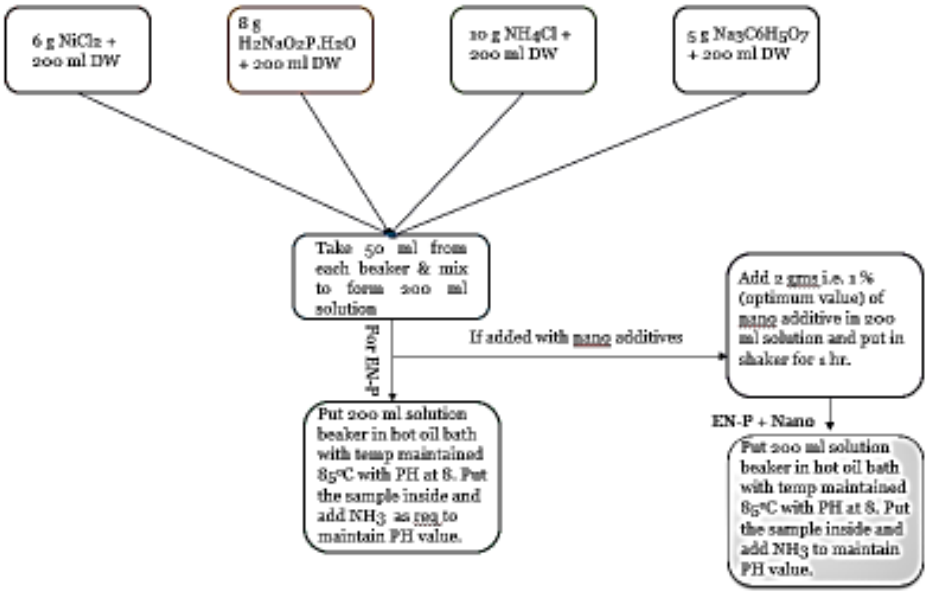


Figure 5:Procedure adopted in preparing Electroless Ni-P bath.

Fig 6: Electroless Ni-P with nano Al2O3Fig 7: Electroless Ni-P coatingFig 8: Electroless Ni-P coating with nano CuO

The amount as shown in table 3 was used to prepare the EN-P bath by measuring the amount of Nickel Chloride (6g/200ml), Sodium hypophosphite (8g/200ml), Sodium tri citrate (5g/200ml) and Ammonium Chloride (10g/200ml). Dissolve the entire components separately with 25 ml of distilled water with PH 7.1 and filtered the dissolved particles. Mixed them together with an addition of 100 ml of distilled water and measured the PH of the bath which should be in range of 8-9 by adding drops of Ammonia Solution to maintain the PH value.

Functionalization of nano materials

- Electroless Ni-P Bath B (Electroless Ni-P + nano additives) has the same procedure of preparing Bath A but only with the addition of nano additives of 1 % i.e. 2 g/200ml (optimum value).
- Electroless Ni-P coating is developed by the auto-catalytic reaction of nickel ions employing sodium hypophosphite as RA. When sodium hypophosphite is used as a RA in EN plating, a typical composition for the deposit is formed ranging from 92 to 95 % nickel and 6 to 9% medium concentration of phosphorus.
- Medium phosphorus deposits provide better corrosion resistance which is the primary goal but on the other hand considerable wear properties in the as- deposited condition would naturally be attributed to greater hardness.
- Nano coated (size 40 nm) materials was produced by shrinking the material at the molecular level to form a denser product. It worked at high surface area to volume ratio because of its nano size resulted in better corrosion resistance, better surface finish and high resistance value.

3: RESULTS & DISCUSSION

The preparation of the Bath B has the same procedure of preparing Bath A, but only with the addition of different combination of nano additives separately. Ni-P with 1 % (0.2 g/200 ml) optimum nano additive value gives more uniform surface finish, wear resistance and corrosion rate resistance value.Increasing percentage of nano additive for more than 1 %, surface roughness value increased but does not have much effect on the quality of coating.Nano additive Al2O3 enhanced the deposition of Ni-P on low alloy steel substrate when added in optimum quantity as compared to other nano additives.

To verify the results, following three type of evaluations were conducted

Mechanical Evaluation

Coating thickness test

Coating thickness of all samples are maintained around 16 to 20 µm for EN-P samples and 80 to 120 µm for paint group samples.

Surface roughness test

Surface roughness for both the groups was conducted, result are as under:

Stage I – Paint group

Coating type	Rz in µm	Remarks
Aluminum paint	4.3	
Zinc Paint	3.7	
Organic Paint	2.3	Minimum surface value
Without Coating	4.5	
Aluminum paint with nano ZnO	5	
Zinc Paint with nano ZnO	6	
Organic Paint with nano ZnO	6.2	
Organic Paint with nano CuO	3.8	
Aluminum paint with nano CuO	9	
Zinc Paint with nano CuO	4.8	
Organic Paint with nano Al2O3	9.8	
Zinc Paint with nano Al2O3	4	
Aluminum Paint with nano Al2O3	4.3	

Table 5: Paint group surface roughness values.

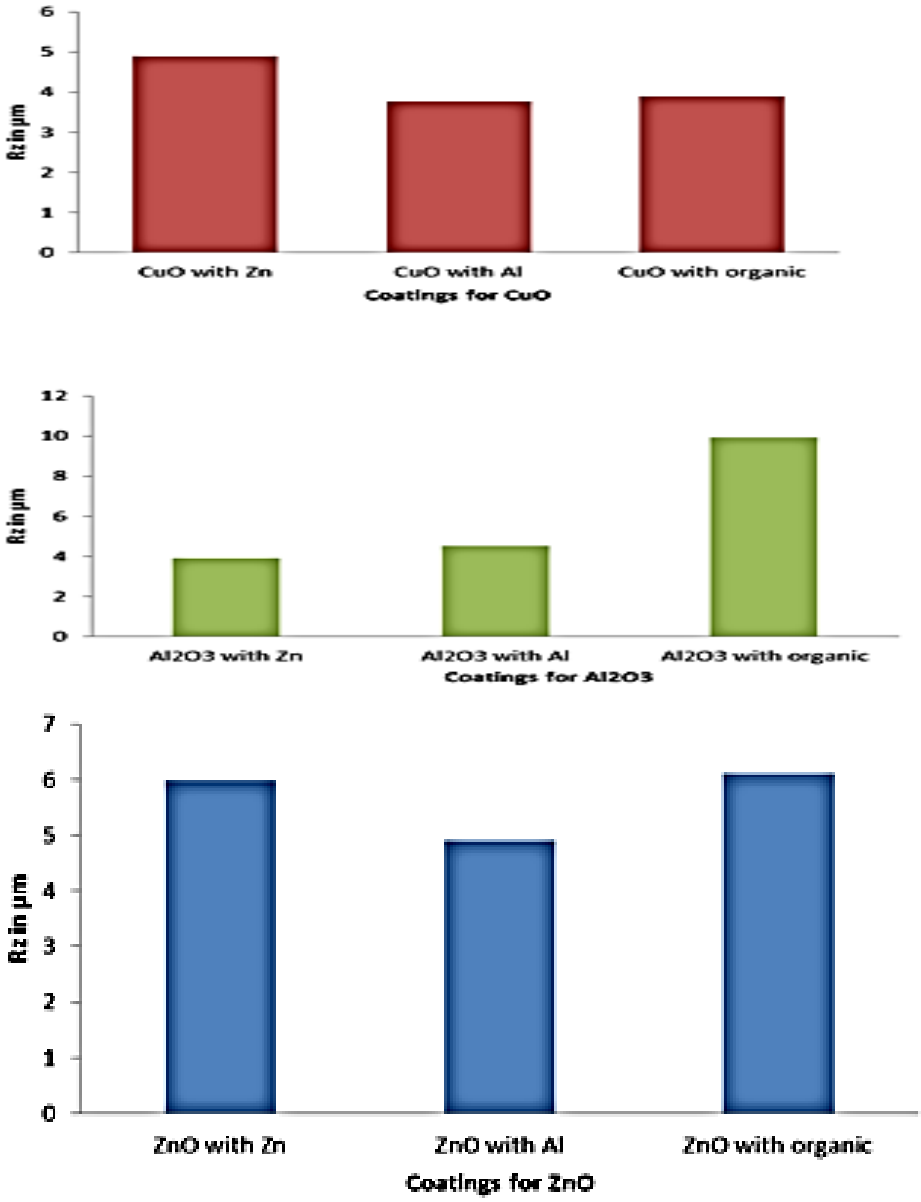


Figure 9: Surface roughness values for paint group with nano additives

Stage II – Electroless Ni-P group

Coating type	Rz in µm	Remarks
Electroless Ni-P	2.1	
Electroless Ni-P with nano CuO	2.2	
Electroless Ni-P with nano ZnO	2.3	
Electroless Ni-P with nano Al2O3	2.12	Minimum surface value

Table 6: Paint group surface roughness values.

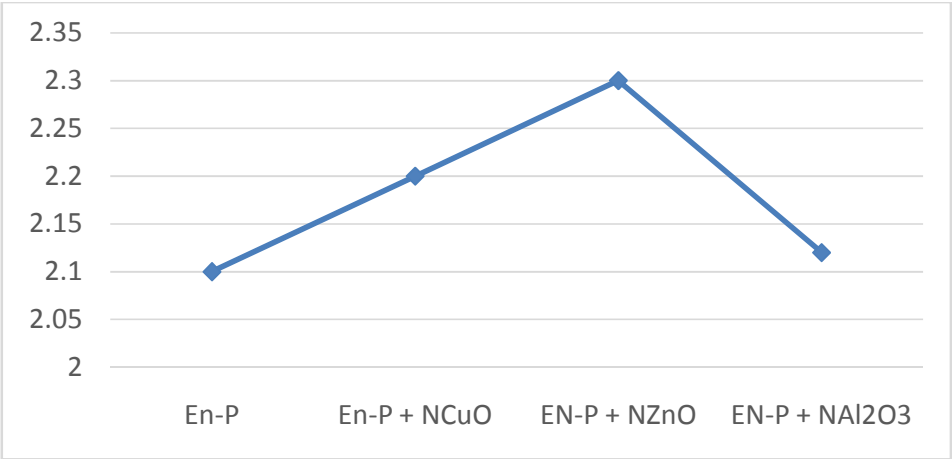


Figure 10: Surface roughness values for Electroless Ni-P group with nano additives

The average roughness value against 1 % concentration of nano additives [7] in both the groups reveals that Electroless Ni-P with nano Al2O3 has least surface roughness value as compared to the organic paint of paint group.

Hardness test

Hardness test for both the groups was also conducted, result are as under:

Stage I & II

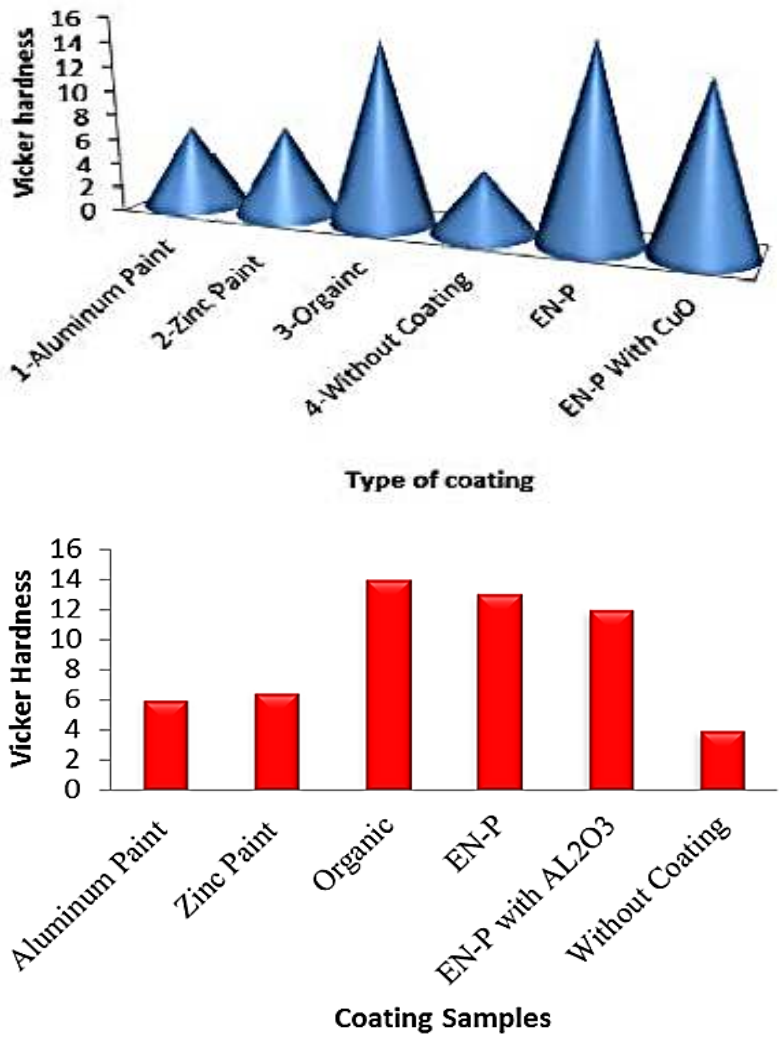


Figure 11: Vicker hardness values for Electroless Ni-P & paint group with nano additives

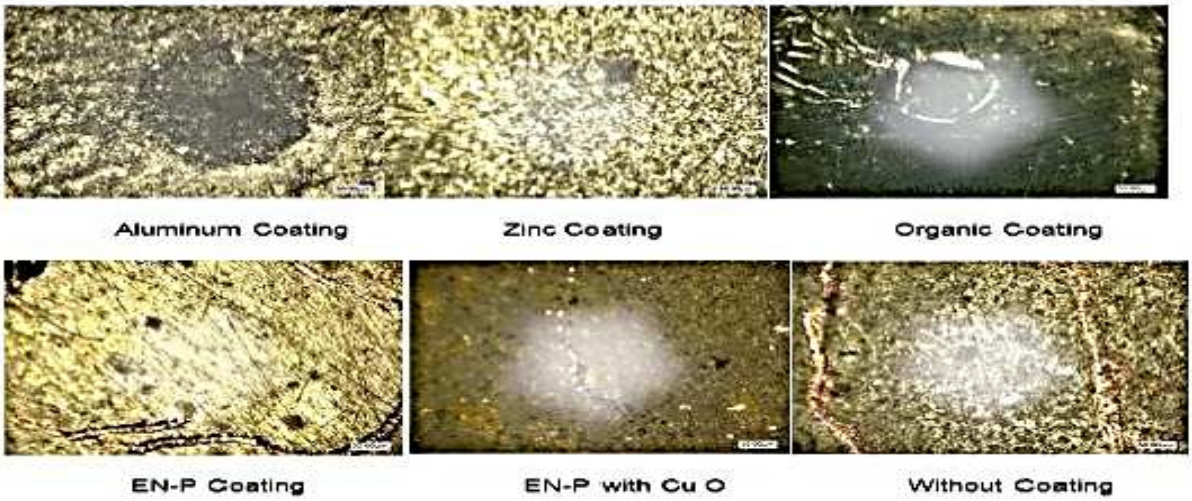


Figure 12: Indent impression for different coatings

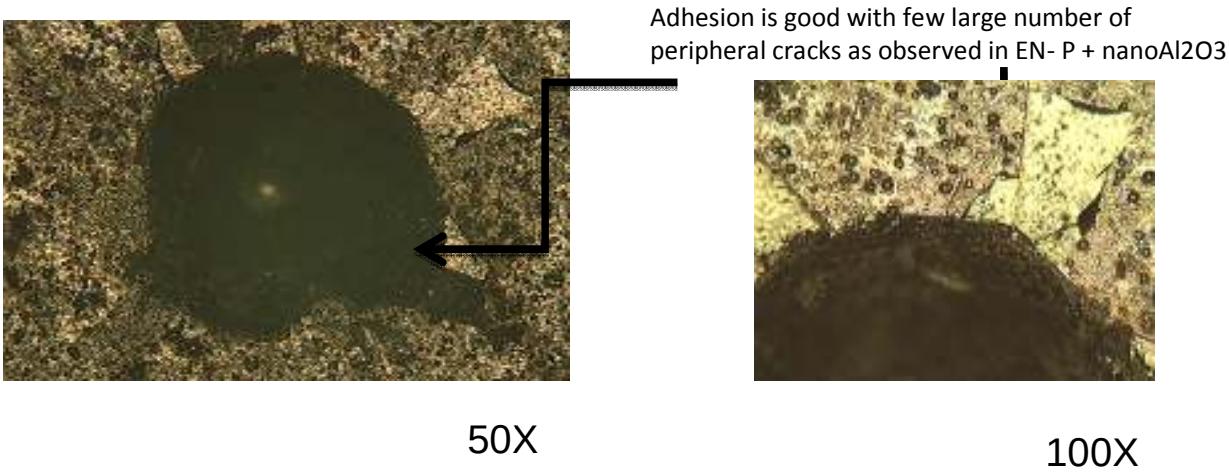


Figure 13: Indent impression for Electroless Ni-P with nano Al2O3

S/No	TYPE OF COATING	Hardness value in VHN200
1	Aluminum paint	150
2	Zinc paint	418
3	Organic paint	110
4	Nano ZnO with organic paint	138
5	Nano ZnO with Aluminum paint	189
6	Nano ZnO with zinc paint	436
7	Nano CuO with organic paint	144
8	Nano CuO with Aluminum paint	193
9	Nano CuO with zinc paint	446
10	Nano Al2O3 with organic paint	143
11	Nano Al2O3 with Aluminum paint	196
12	Nano Al2O3 with zinc paint	439
13	Electroless Ni-P with Nano ZnO	560
14	Electroless Ni-P with Nano Al2O3	595
15	Electroless Ni-P with Nano CuO	573
16	Electroless Ni-P	450

Table 7: VHN value for both the groups

SEM image as shown in figure 13 aboveproved that Electroless Ni-P with nano Al2O3 has less hair line cracks with few delamination as compare to other combinations with highest VHN 200 value as reported in Table 7 above.

Characterization Evaluation

Optical microscope

The optical images for different coatings are given below in figure 14.The dark sides revealed in the figures are areas with oxidation. It seems that Electroless Ni-P with nano Al2O3 has few oxidized areas. Moreover, it also seems that the surface texture of the same is also very smooth as compared to other coating image

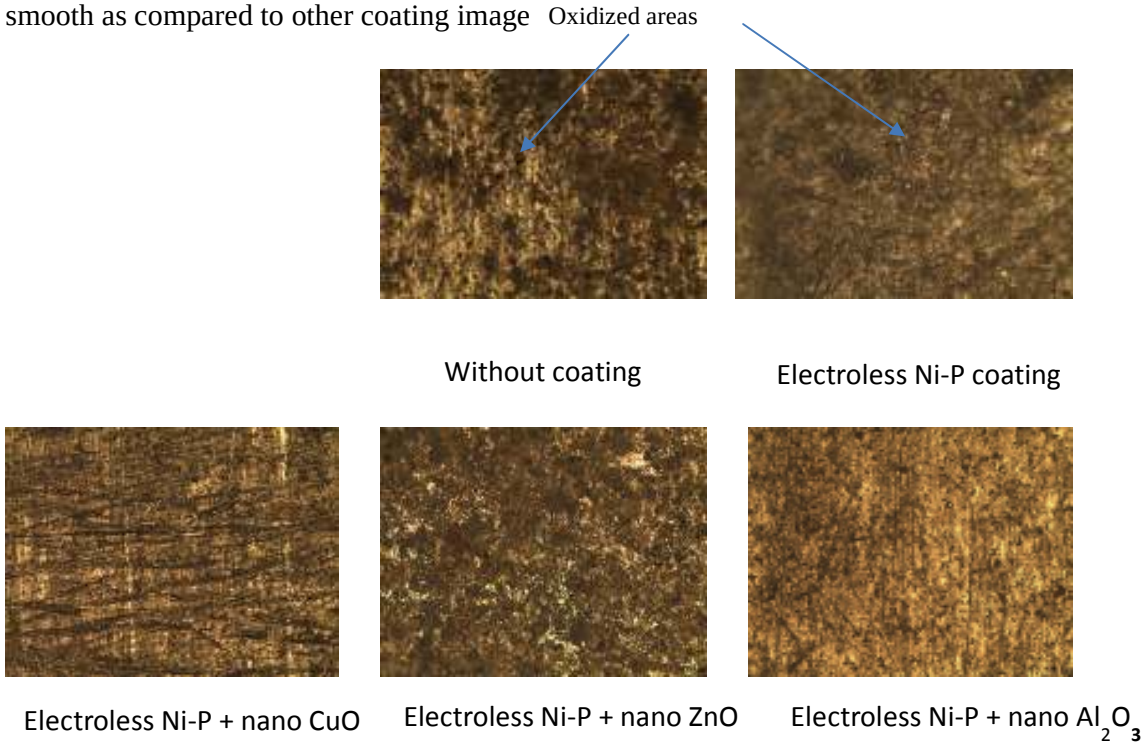


Figure 14: Optical images of the coatings

Scanning Electron Microscope

SEM images as shown in figure 15 and 16 proves that Aluminum coating,Zinc coating and organic coating has rough surface finish as compared to Electroless Ni-P with nano Al2O3.It also demonstrates the deposition of Nickel with small particles of Nano Al2O3 that contributes better resistance than sample coated with Electroless Ni-P only.

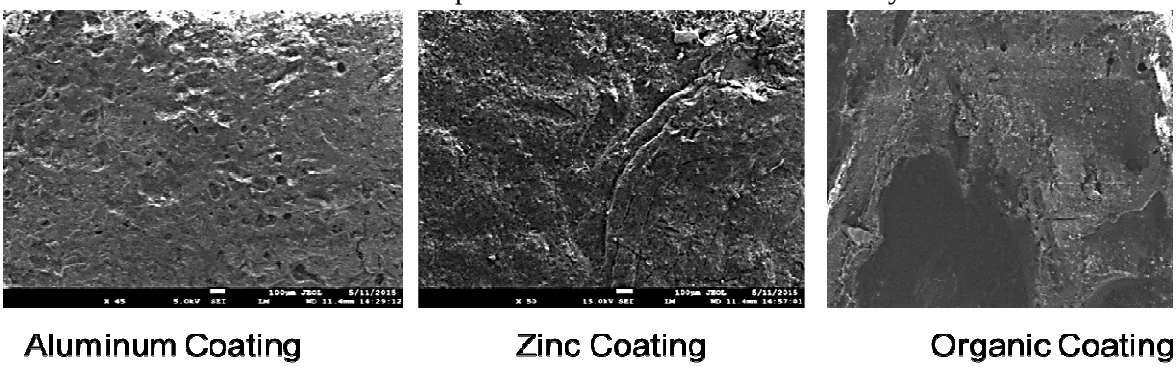
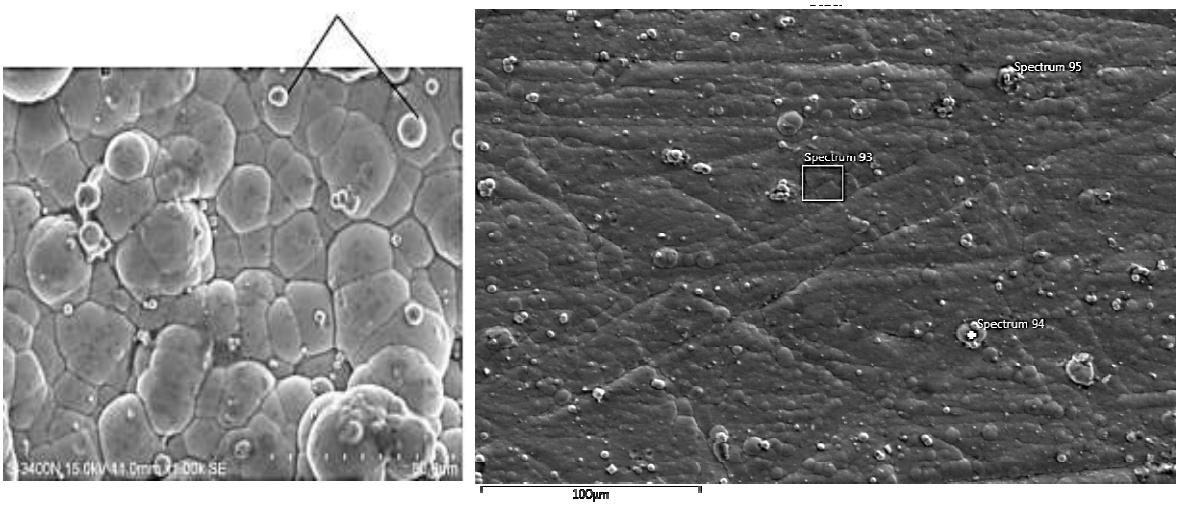


Figure 15: SEM images of the coatings

Nano Al2O3



1000 X

Figure 16: SEM image of EN-P with nano Al2O3

X-ray Diffraction

The percentage of elements present in the coatings are shown in figure 17 and 18.

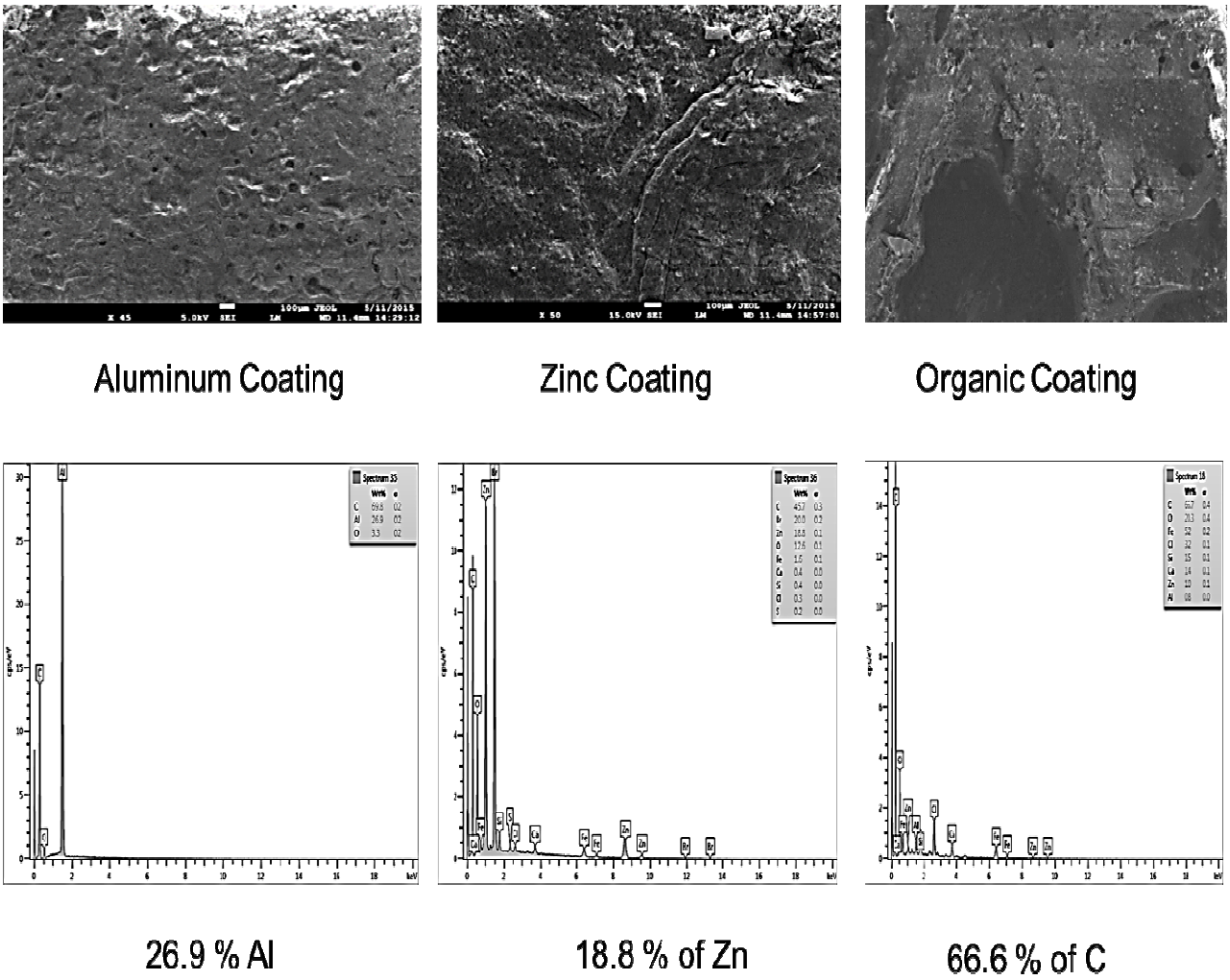


Figure 17: XRD image of coatings [8]

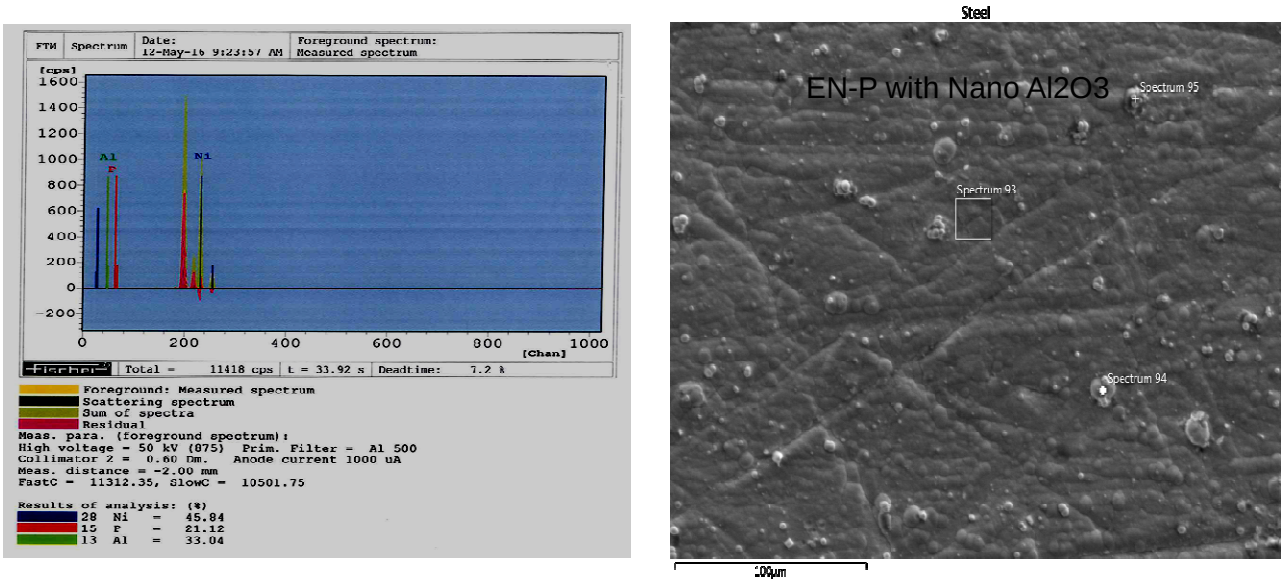


Figure 18: XRD image of EN-P with Nano Al2O3 coating

Corrosion Resistance Evaluation

To evaluate the corrosion resistance of all the samples three type of corrosion resistance test were conducted.

Atmospheric exposure test

The test carried out for 58 days in ambient conditions of Oman Muscat where temperature varied from 25 to 46 0C with relative humidity ranging from 60 to 65 % and wind speed of 3 m/s. Results of the test for both the groups are shown in table 8 below. The corrosion resistance values in millimeter per year (mmpy) were calculated from the basic weigh loss formula [10].

$$\text{Corrosion rate in mmpy} = 84\ (W/DAT)$$

Where

W = Weight loss in grams

D = Density of low alloy steel A1031B,7.874 gms / cm3 [10]

A = Area of sample,59 cm2

T= Time of exposure,1400 hours.

From table 8 it is evident that Electroless Ni-P with nano Al2O3 reported highest corrosion resistance of 0.0353 as compared to other samples.

S/No	TYPE OF COATING	Weight loss (gms)	Atmospheric Exposure test mmpy
1	Aluminum paint	1.26	0.1651
2	Zinc paint	5.04	0.6603
3	Organic paint	6.05	0.7926
4	Nano ZnO with organic paint	0.89	0.215
5	Nano ZnO with Aluminum paint	0.8	0.1932
6	Nano ZnO with zinc paint	0.79	0.1908
7	Nano CuO with organic paint	0.6	0.1949
8	Nano CuO with Aluminum paint	0.2	0.0483
9	Nano CuO with zinc paint	0.56	0.1353
10	Nano Al2O3 with organic paint	0.68	0.1643
11	Nano Al2O3 with Aluminum paint	0.18	0.0453
12	Nano Al2O3 with zinc paint	0.2	0.0483
13	Electroless Ni-P with Nano ZnO	0.21	0.0412
14	Electroless Ni-P with Nano Al2O3	0.18	0.0353
15	Electroless Ni-P with Nano CuO	0.24	0.0471
16	Electroless Ni-P	0.43	0.0843

Table 8: Atmospheric Exposure test

Dry / wet test

The dry wet test was also conducted for 14 days in sea water maintaining the PH value of 7.6 during the test. Ambient room temperature was maintained around 22 to 25 0C with relatively low humidity. Samples were deposited in the sea water for 16 hours and then dry under same conditions of 8 hours.This process continued for 14 days. Results are shown in table 9 below using same corrosion rate formula as discussed above.

W = Weight loss in grams

D = Density of low alloy steel A1031B, 7.874 gms / cm3

A = Area of sample, 13.6 cm2

T= Time of exposure, 336 hours.

S/No	TYPE OF COATING	Weight loss (gms)	Dry/Wet test mmpy
1	Aluminum paint	0.576	1.4
2	Zinc paint	1.10	2.679
3	Organic paint	1.90	4.628
4	Nano ZnO with organic paint	2.31	5.62
5	Nano ZnO with Aluminum paint	1.14	2.78
6	Nano ZnO with zinc paint	1.61	3.92
7	Nano CuO with organic paint	0.3	0.73
8	Nano CuO with Aluminum paint	2.2	5.45
9	Nano CuO with zinc paint	0.942	2.29
10	Nano Al2O3 with organic paint	0.391	0.95
11	Nano Al2O3 with Aluminum paint	0.139	0.34
12	Nano Al2O3 with zinc paint	0.209	0.51
13	Electroless Ni-P with Nano ZnO	0.019	0.0481
14	Electroless Ni-P with Nano Al2O3	0.0169	0.0412
15	Electroless Ni-P with Nano CuO	0.0240	0.0584
16	Electroless Ni-P	0.0316	0.077

Table 9: Dry / wet test

From the above table 9 it is reported that Electroless Ni-P with nano Al2O3 has highest corrosion resistance value of 0.0412 mmpy as compared to other combinations.

Potentio-stat test

To identify the corrosion rate, a technique is used in which anodic and cathodic behavior of the metal surface were checked using electrolytic solution like sea water or acidic solution. The coated sample is called working electrode (WE) and the reference electrode (RE) should be typically a stable material which is used to monitor and maintain potential under WE surface.The formula used to calculate corrosion current is

mmpy = 3.27 x 10-3 x Icorr x W / r [11]

Where

Icorr = Current in Teffal graph in µA/cm2

W = Equivalent weight in grams for low alloy steel A103B.

r = Material density, 7.874 gms / cm3

S/No	TYPE OF COATING	Weight loss (gms)	Icorr µA/cm2	Potentio stat test mmpy
1	Aluminum paint	1.1	1.05	0.482
2	Zinc paint	1.27	1.1	0.583
3	Organic paint	1.16	1.2	0.637
4	Nano ZnO with organic paint	0.523	4.2	0.914
5	Nano ZnO with Aluminum paint	0.461	4.7	0.9
6	Nano ZnO with zinc paint	0.0678	3.8	0.107
7	Nano CuO with organic paint	0.0418	4.6	0.08
8	Nano CuO with Aluminum paint	0.0431	4.4	0.079
9	Nano CuO with zinc paint	0.0419	4.7	0.082
10	Nano Al2O3 with organic paint	0.0316	5.4	0.071
11	Nano Al2O3 with Aluminum paint	0.0302	5.5	0.069
12	Nano Al2O3 with zinc paint	0.0346	5.0	0.072
13	Electroless Ni-P with Nano ZnO	0.0281	7.2	0.0834
14	Electroless Ni-P with Nano Al2O3	0.0288	6.2	0.0718
15	Electroless Ni-P with Nano CuO	0.0279	6.5	0.0754
16	Electroless Ni-P	0.415	6.4	0.0742

Table 10: Potentio Stat test

From the current values of I corr and weight loss it is reported that Electroless Ni-P with nano Al2O3 provided with maximum corrosion resistance of 0.0718 mmpy as compared to other combinations at ambient temperature of around 25 0C with relatively low humidity. The Tefal graph results for few samples are also shown in figure 19 and 20.

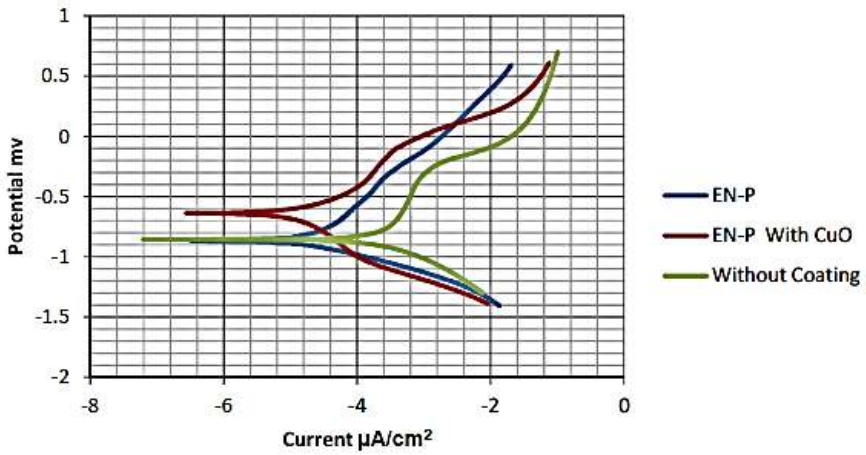


Figure 19:Potentio Stat test for few samples

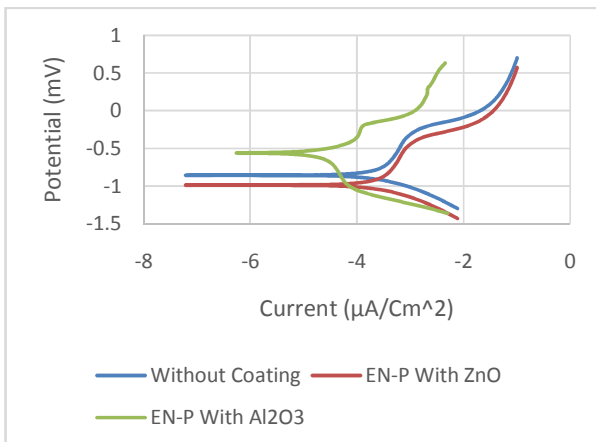


Figure 20: Potentio Stat test for Electroless Ni-P samples

Coating efficiency

For Ni recovery NiCl2 is 30 gms / liter

Similarly for 200 ml it is 6 gms / 200 ml

Therefore in aqueous bath Nit = 2.71 gms, [13]

Sample weight before coating = 37.53 gms

Sample weight after coating = 38.20 gms

Weight of coating Nip = 38.20 – 37.53 = 0.67 gms.

Coating Efficiency = $\frac{\mathbf{Nip}}{\mathbf{Nit}}$= 0.67 / 2.71 = 0.2472 = 24.72 %

It means only 24.72% of nickel is deposited on the substrate rest 72.58 % is wasted in electrolyte.

Benefits of using nano additives

a. Bath B (Electroless Ni-P + nano additives) has the same procedure of preparing Bath A, but only with the addition of nanoadditives of 1 % i.e. 2 g/200ml (optimum value).

b. Electroless Ni-P coating is developed by the auto-catalytic reaction of nickel ions employing sodium hypophosphite as RA. When sodium hypophosphite is used as a RA in Electroless Ni-P plating, a typical composition for the deposit is formed ranging from 92 to 95 % nickel and 6 to 9% medium concentration of phosphorus.

c. Medium phosphorus deposits provide better corrosion resistance which is the primary goal but on the other hand considerable wear properties in the as- deposited condition would naturally be attributed to greater hardness.

d. Nano coated (size 40 to 50 nm) materials are produced by shrinking the material at the molecular level to form a denser product. It works at high surface area to volume ratio because of its nano size resulted in better corrosion resistance, better surface finish and high resistance value.

e. A comparison with Electroless Ni-P coating with nano Al2O3 can be shown below the benefit of adding the nano additive. Take values of atmospheric exposure test from table 11 to calculate the percentage reduction in corrosion rate.
% reduction of corrosion rate =
(Corrosion rate for Electroless Ni-P – Corrosion rate for Electroless Ni-P) + nano Al2O3
÷ Corrosion rate for
En-P

$$= 0.0843 – 0.0353 \div 0.0843 = 58.12\ \%$$

Corrosion rate of Electroless Ni-P with nano Al2O3 is	0.0353 mmpy
Electroless Ni-P with nano ZnO is	0.0412 mmpy
Electroless Ni-P with nano CuO is	0.0471 mmpy.

After adding Electroless Ni-P with nano Al2O3, corrosion rate is improved by 58.12 % as compared to without of nano particles.

Similarly corrosion rate for EN-P in comparison with organic paint is	89.40 %
Corrosion rate for EN-P with nano CuO in comparison with organic paint is	94 %
Corrosion rate for EN-P with nano ZnO in comparison with organic paint is	94.8 %
Corrosion rate for EN-P with nano Al2O3 in comparison with organic paint is	95.5 %

f. The observed results also reported that the corrosion rate of Electroless Ni-P with nano Al2O3 is 0.0353 mmpy followed by Electroless Ni-P with nano ZnO is 0.0412 mmpy anElectroless Ni-P EN-P with nano CuO is 0.0471 mmpy. After adding Electroless Ni-P with nano Al2O3, corrosion rate is improved by 58.12 % as compared to without addition of nano particles.

g. Similarly if corrosion rate for Electroless Ni-P is compared in comparison with organic paint is 89.40 %.Corrosion rate for Electroless Ni-P with nano CuO in comparison with organic paint is 94 %.Corrosion rate for Electroless Ni-P with nano ZnO in comparison with organic paint is 94.8 % and corrosion rate for Electroless Ni-P with nano Al2O3 in comparison with organic paint is 95.5 % which gives the highest value as compared to other comparative values.

Surface Roughness of Nano additives

The variation of average surface roughness value of the coated layer of different combinations are summarized in table 11 below. The average surface roughness values of nano additives are low as compared to other coated combinations.

Micro hardness of Nano additives

Table 11 shows an improvement in of microhardness of Electroless Ni-P coating with respect to nano Al2O3, followed with nano CuO and then with nao ZnO. The reason for increase in the microhardnessvalues of Electroless Ni-P coatings with nano additives may be due to uniform deposition of nanoparticles on the Ni-P matrix and filling the micro gaps of Ni–P coated layer thus increasing the density of coated layer. Similar kind of trend was obtained by earlier researchers after adding nano additives in their respective studies.

S/No	TYPE OF COATING	Surface Roughness Rz µm	Hardness value in VHN200
1	Aluminum paint	4.29	150
2	Zinc paint	3.77	418
3	Organic paint	2.32	110
4	Nano ZnO with organic paint	6.25	138
5	Nano ZnO with Aluminum paint	5.12	189
6	Nano ZnO with zinc paint	6.05	436
7	Nano CuO with organic paint	4.0	144
8	Nano CuO with Aluminum paint	3.8	193
9	Nano CuO with zinc paint	4.9	446
10	Nano Al2O3 with organic paint	9.2	143
11	Nano Al2O3 with Aluminum paint	4.25	196
12	Nano Al2O3 with zinc paint	4.05	439
13	Electroless Ni-P with Nano ZnO	2.3	560
14	Electroless Ni-P with Nano Al2O3	2.12	595
15	Electroless Ni-P with Nano CuO	1.9	573
16	Electroless Ni-P	2.1	450

Table 11: Surface roughness & micro hardness of groups

Comparative performance

Table 12 reported comparative performance of mechanical as well as corrosion resistance evaluation. Electroless Ni-P with nano Al2O3 showed best performance in terms of mechanical as well as corrosion resistance evaluation. Comparative performances for each group are also presented in graphical mode from figure 21 to 24.

S/No	TYPE OF COATING	Mechanical Evaluation		Corrosion Resistance Evaluation		
		Surface Roughness Rz µm	Hardness value in VHN200	Atmospheric Exposure test mmpy	Dry/Wet test mmpy	Potentiostat test mmpy
1	Aluminum paint	4.29	150	0.1651	1.4	0.125
2	Zinc paint	3.77	418	0.6603	2.679	0.583
3	Organic paint	2.32	110	0.7926	4.628	0.637
4	Nano ZnO with organic paint	6.25	138	0.215	5.62	0.914
5	Nano ZnO with Aluminum paint	5.12	189	0.1932	2.78	0.9
6	Nano ZnO with zinc paint	6.05	436	0.1908	3.92	0.107
7	Nano CuO with organic paint	4.0	144	0.1949	0.73	0.08
8	Nano CuO with Aluminum paint	3.8	193	0.0483	5.45	0.079
9	Nano CuO with zinc paint	4.9	446	0.1353	2.29	0.082
10	Nano Al2O3 with organic paint	9.2	143	0.1643	0.95	0.071
11	Nano Al2O3 with Aluminum paint	4.25	196	0.0453	0.34	0.069
12	Nano Al2O3 with zinc paint	4.05	439	0.0483	0.51	0.072
13	EN-P with Nano ZnO	2.3	560	0.0412	0.0481	0.0834
14	EN-P with Nano Al2O3	2.12	595	0.0353	0.0412	0.0718
15	ENP with Nano CuO	1.9	573	0.0471	0.0584	0.0754
16	EN-P	2.1	450	0.0843	1.047	0.0742

Table 12: Comparative performance of groups

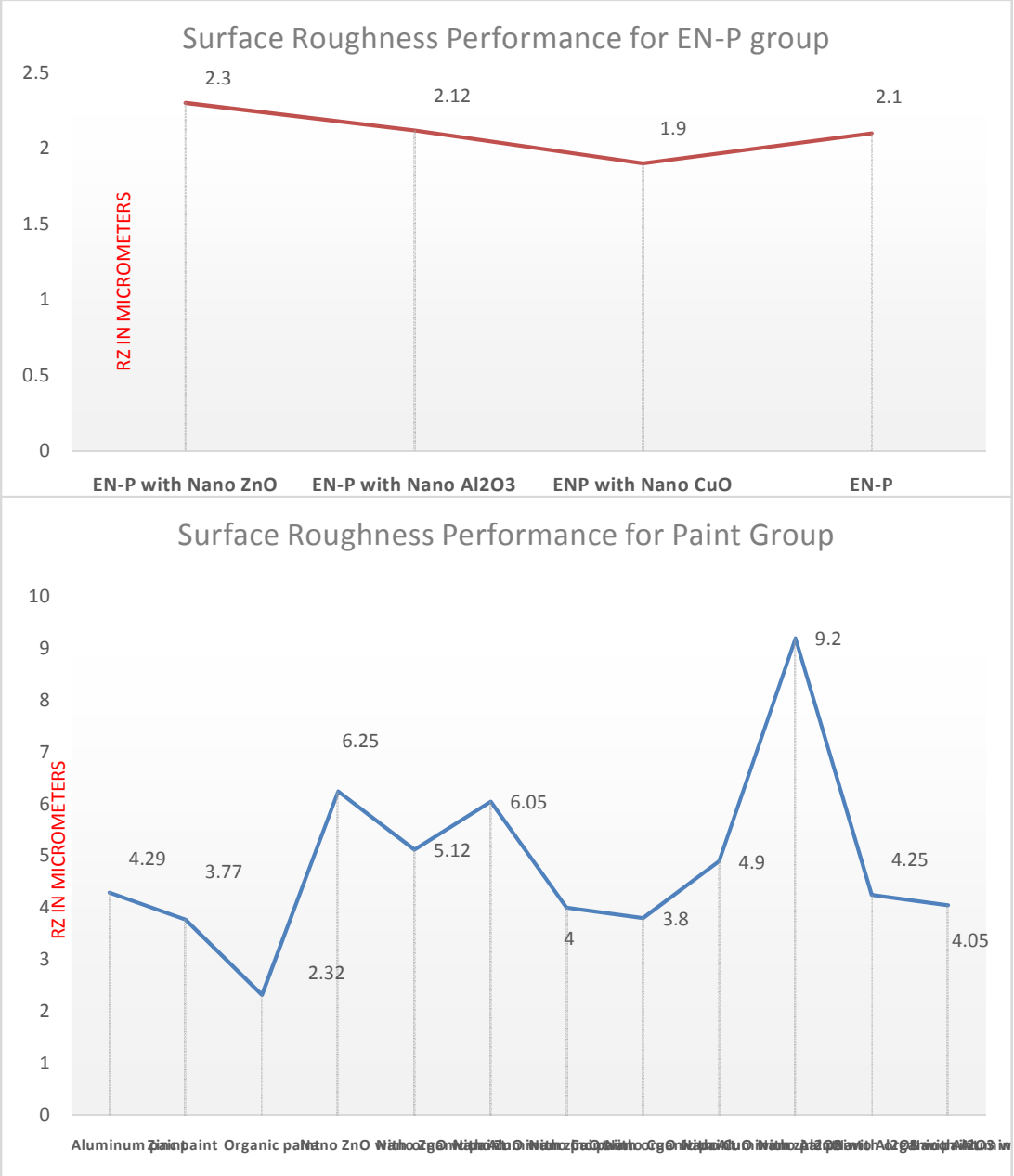
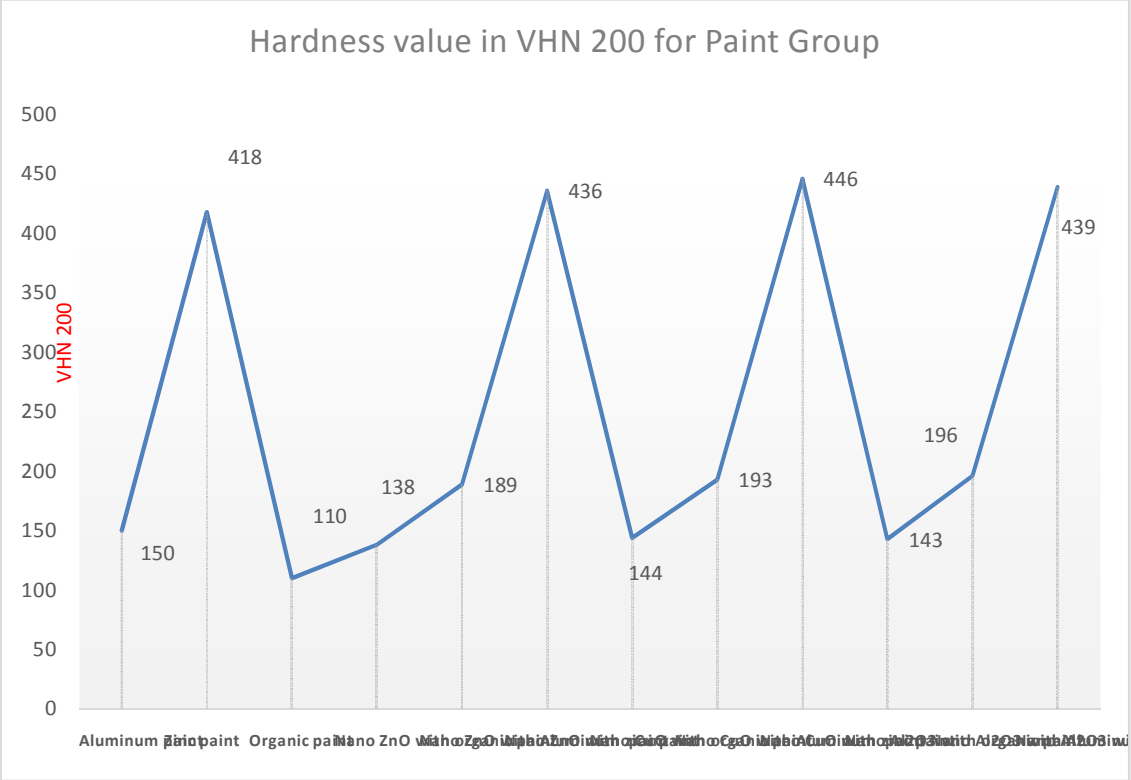


Figure 21: Surface roughness performance for both groups

To have performance comparison for Electroless Ni-P with nano Al2O3 for 2.12 µm Rz value in comparison with Aluminum paint of 4.29 µm Rz value the reported surface roughness improvement is 50.5%. By addition of nano Al2O3 in Electroless Ni-P bath around 50% surface roughness is improved as compared to ordinary Aluminum paint.



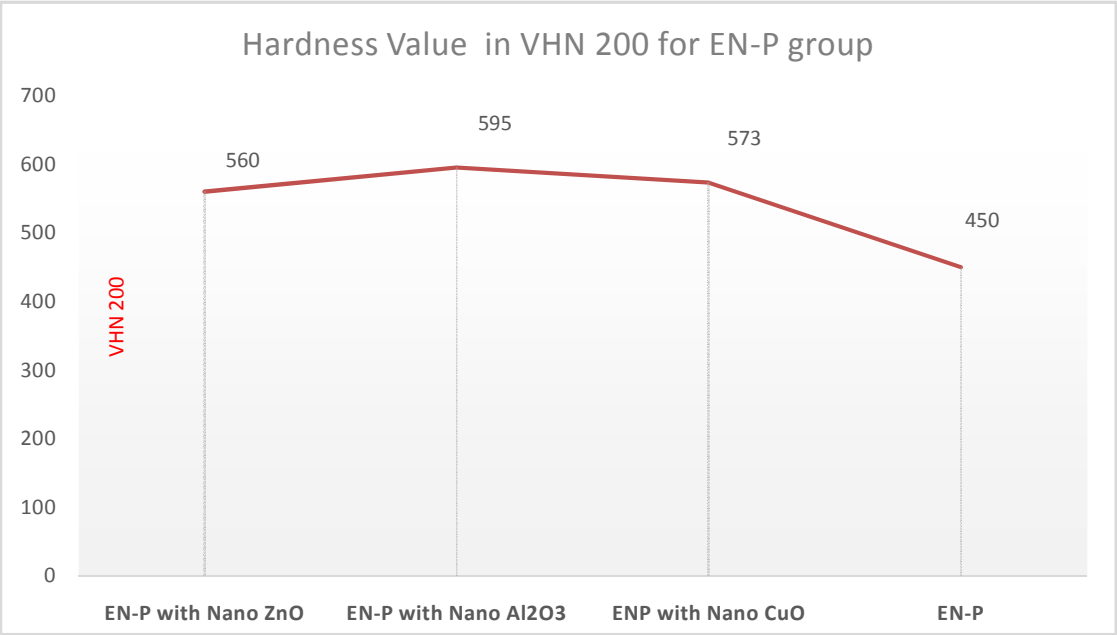


Figure 22: Micro hardness VHN200 performance for both groups

To have performance comparison for Electroless Ni-P with nano Al2O3 of 595 VHN 200 value in comparison with Aluminum paint of 150 VHN 200 value the reported microhardness improvement is 75 %. By addition of nano Al2O3 in Electroless Ni-P bath around 75 % hardness is improved as compared to ordinary Aluminum paint.

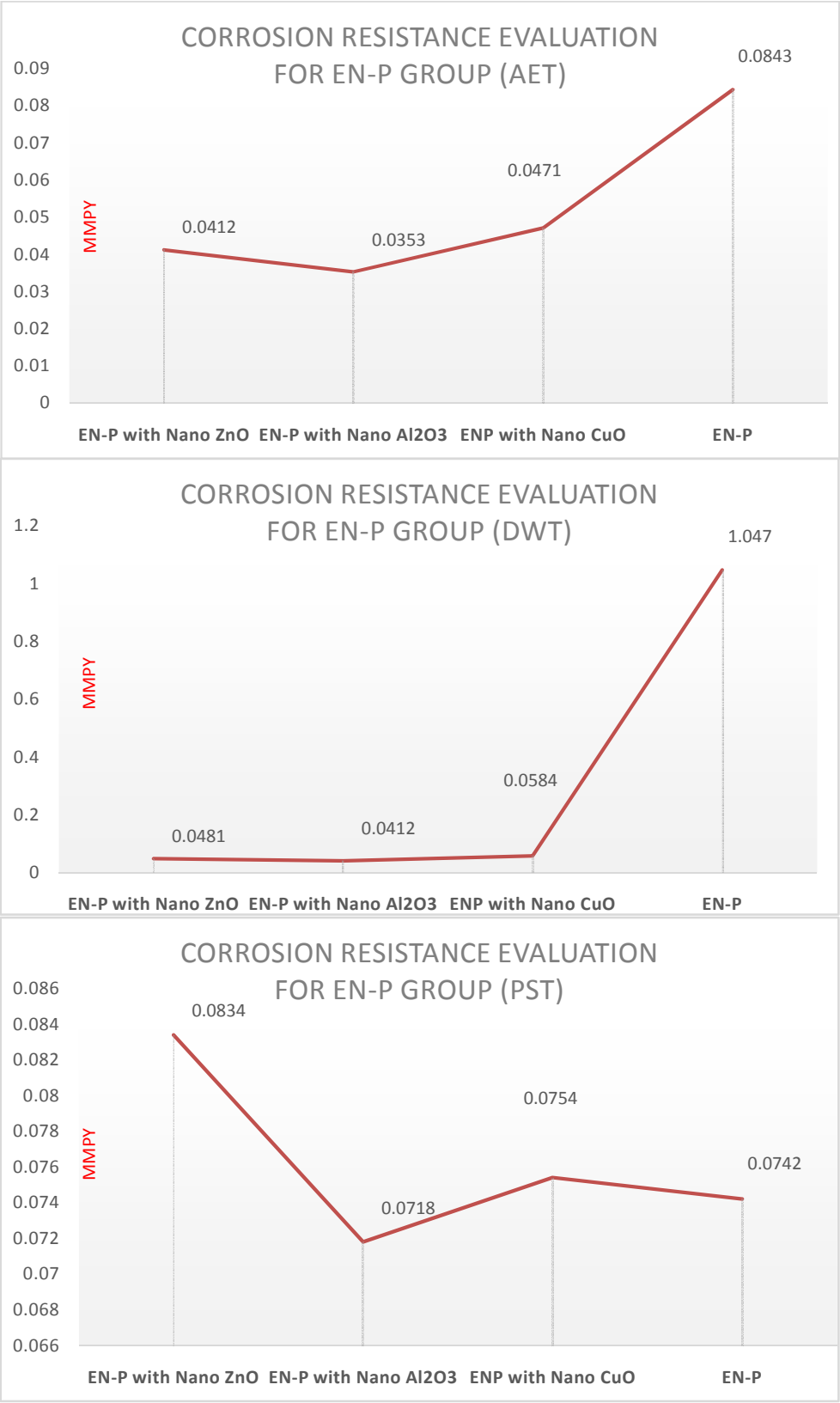
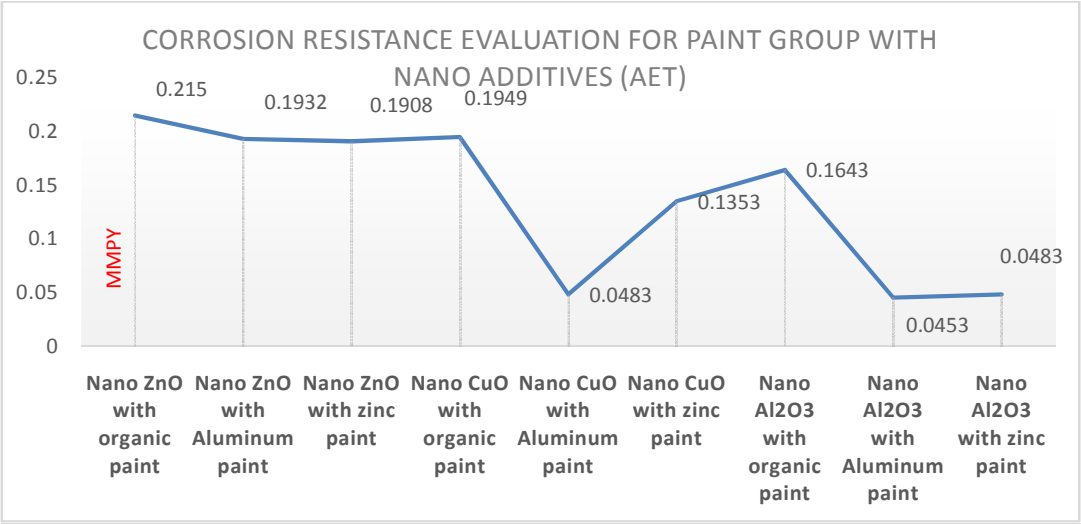
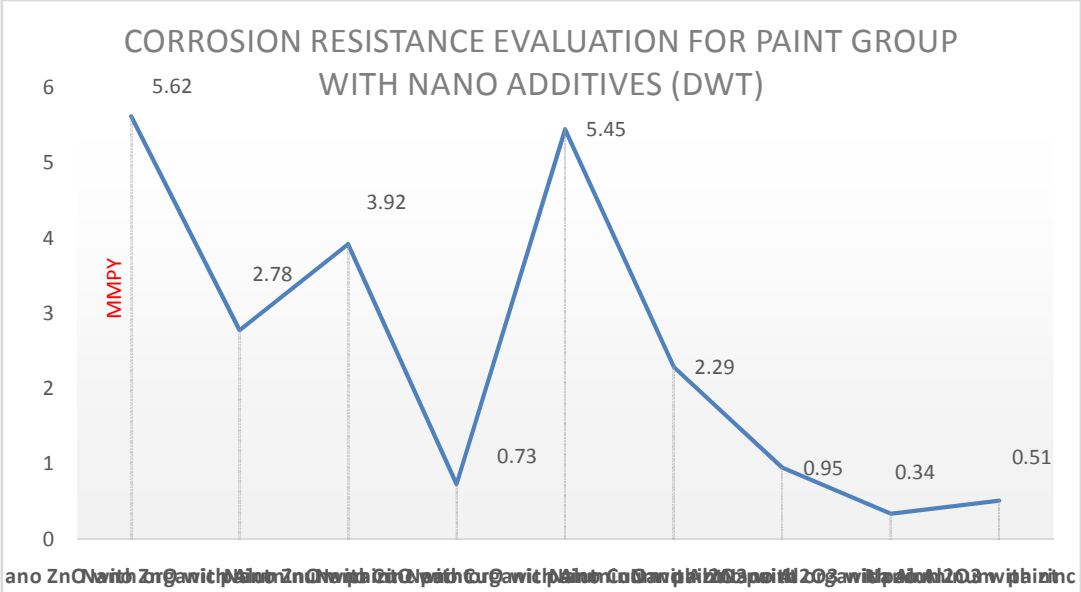


Figure 23: Corrosion resistance performance for EN-P group





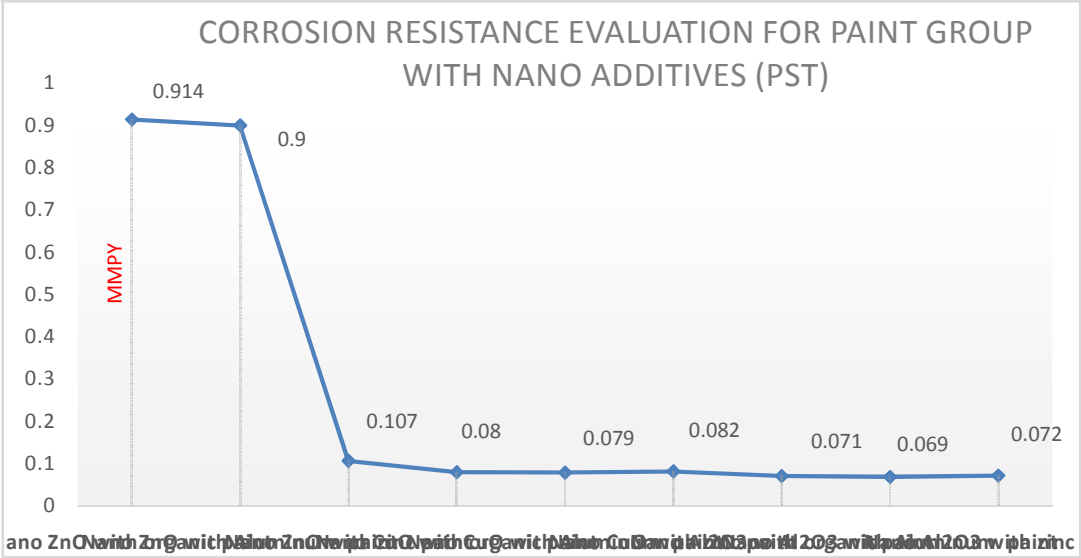


Figure 24: Corrosion resistance performance for Paint group

4: CONCLUDING REMARKS

A comprehensive experimental study under specific coating conditions on the influence of addition of various nano additives with in the Electroless Ni-P bath on the mechanical properties and tribological properties of the coatings produced has been carried out and the results are presented and analyzed (table 12,figure 21 to 24). In general, it has been observed that the surface finish, micro hardness and corrosion resistance of the EN-coated layers improved significantly with the addition of nano additives. Based on the present investigation, the following specific conclusions could be drawn:

a. 37 samples were coated and investigated under paint and Electroless Ni-P group. These samples were examined under mechanical, characterization and corrosion resistance evaluation. Refer to the comparative performance of the coatings as shown in table 12, it is reported that 1 % concentration of nano additives in Electroless Ni-P bath gives better performance results as compare to 0.5, 1.5 and 2 % concentrations.Electroless Ni-P with nano Al2O3 results under corrosion resistance, micro hardness and surface finish values reported better results as compared toElectroless Ni-P with Nano CuO followed by Electroless Ni-P with nano ZnO.

b. There was an improvement in the surface finish of around 38% due to addition of nano additives of Al2O3 in EN bath refer figure 21. Addition of 1 % optimum value of nano additive to the Electroless Ni-P bath prevents the floatation of nickel particles generated during the chemical reaction of the coating process. Since the nickel particles do not float and move to the top surface of bath, more percentage of nickel particles get deposited as a fine layer thus improving the surface finish.

c. Considering the investigation against atmospheric exposure test, dry / wet test and potentio stat test, the results reflected that lowest corrosion rate is for Electroless Ni-P with nano AL2O3 and highest corrosion rate is for organic paint group refer figure 23 and 24.

d. In the EN-P the result investigates that by the addition of nano Al2O3, gaps in Electroless Ni-P deposition with nano additives reduces the corrosion rate occurrence.

e. Mirohardness of the samples was also studied through Vicker hardness. The VHN 200 for EN-P with addition of Al2O3 is maximum as compared to other combinations in both the groups refer figure 22. The reason for increase in the microhardness value of Electroless Ni-P coating with nano additives may be due to uniform deposition of nanoparticles on the Ni-P matrix and filling the micro gaps of Ni–P coated layer thus increasing the density of coated layer. Similar research work was also reported by earlier researchers after adding nano additives in their research work.

f. Surface morphology of the samples was studied through SEM to analyze the microstructure of the coatings. The image of EN depicts that the nickel was deposed on the surface of the sample. Image of EN-P with addition of Al2O3 shows the deposition of Nickel with small particles of Nano Al2O3 that contributes better resistance than sample coated with Electroless Ni-P only. Moreover, the sample without coating shows the surface area totally corroded.

h. Coating efficiency is reduced to 24.75 % only for Electroless Ni-P coated samples, however,75.28% nickel was wasted in electrolyte.

i. This work may be further continued in future trying out with other nano additives and surfactants [13] to improve the coating efficiency, corrosion rate, surface roughness, hardness and wear.

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