



Electroless Ni–P–SnO₂ and Ni–P–Mn₂O₃ Composite Coatings on AH36 Marine Steel

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ABSTRACT

Electroless coating offers a promising method for enhancing the properties of structural materials such as AH36 steel by applying protective surface layers that improve corrosion resistance and mechanical durability. This study investigates the development and evaluation of electroless nickel–phosphorus–tin(IV) oxide (Ni–P–SnO₂) and nickel–phosphorus–manganese(III) oxide (Ni–P–Mn₂O₃) composite coatings incorporating manganese oxide and tin oxide nanoparticles synthesized via a precipitation method. Optical microscopy and SEM analyses revealed a uniform and pore-free coating distribution across the substrate. EDS identified the elemental composition of the deposits, confirming the presence and uniform dispersion of nickel, Mn₂O₃, and SnO₂ nanoparticles. Mn₂O₃ reinforcement significantly improved microhardness (up to 545 HV). Microhardness tests indicated a substantial increase in hardness of 14.6% for the coated AH36 steel compared to the untreated material, highlighting its enhanced mechanical performance. AFM imaging demonstrated smoother surface topography and reduced roughness (126% improvement) in the coated samples, suggesting improved surface quality and potential for lower friction in practical applications. Scratch resistance tests further validated the durability of the coated surface, making it suitable for demanding environments. The corrosion resistance of the Ni–P-coated substrate improved significantly by 16.4% compared to the uncoated substrate.

Keywords: Electroless coating; AH36 steel; Ni–P–Mn₂O₃; Ni–P–SnO₂; Mechanical properties; Corrosion resistance; Marine applications.

1. INTRODUCTION

AH36 marine-grade steel is a high-strength, low-alloy structural steel widely used in shipbuilding due to its excellent toughness, weldability, and load-bearing capacity under harsh marine environments. Electroless Ni–P coatings provide a uniform, autocatalytic nickel–phosphorus layer that significantly enhances the corrosion resistance, wear resistance, and surface hardness of AH36 steel without requiring external electrical current. The importance of naval steel, particularly high-strength AH36 steel, in maritime applications is well established. Continuous efforts to enhance corrosion resistance are essential for prolonged durability in harsh marine environments (Araghi and Paydar, 2013). Electroless coating proves promising, creating a uniform, protective layer on metallic surfaces. These coatings improve surface hardness, extend steel lifespan, and reduce maintenance and operational costs. Besides efficiency gains, the environmental impact is positive, contributing to the sustainability of the naval operation (Brenner and Riddel, 1953). When nanoparticles or microparticles are introduced into a binary matrix, the system is termed a composite coating.

During the coating process, the particles are incorporated onto the substrate and co-deposited with Ni–P, forming a composite coating (Frank *et al.* 2020).

SnO₂ and Mn₂O₃ nanoparticles were synthesized to enhance the corrosion resistance and mechanical strength of electroless Ni–P coatings in chloride-rich marine environments. Their controlled nano-size and uniform dispersion in the Ni–P matrix improve hardness, surface smoothness, and overall durability of AH36 steel. Tin dioxide (SnO₂) provides a versatile means of improving the tribological and mechanical properties of steel when incorporated into composite coatings. Enhanced hardness from incorporating SnO₂ into steel confers improved resistance to wear and deformation, which is particularly important for durability in various situations (Radha and Sreekanth, 2020). SnO₂ is an adaptable way to increase steel's lifespan and performance in various industrial applications because of these combined effects (Mahmoud and Fouad, 2015). Nickel–manganese alloys are a class of alloys composed of nickel and manganese. The properties of these alloys can vary depending on the specific composition and processing conditions. Nickel–

manganese alloys may exhibit ferromagnetic properties, and the magnetic behavior can be influenced by the ratio of nickel to manganese (Shi *et al.* 2021). Nickel–manganese alloys can have improved corrosion resistance compared to other nickel alloys (Anzini *et al.* 2020). The mechanical properties of nickel–manganese alloys depend on factors such as heat treatment, alloy composition, and grain structure (Cheng *et al.* 2025). Nickel–manganese alloys may have good stability at high temperatures, making them suitable for specific applications (Wendler *et al.* 2013). The nanoparticles entered into the Ni–P matrix without forming any porous gaps in the coating. The precipitation of Ni–Mn alloys and SnO₂ involves the formation of solid particles or crystals from a solution or a gaseous phase. Precipitation of Ni–Mn alloys: A common method involves preparing a solution containing nickel and manganese salts. Precipitation can be induced by adding a precipitating agent, such as a base (e.g., sodium hydroxide), to the solution. The resulting precipitate is then collected and further processed to obtain the desired Ni–Mn alloy (Bastami and Entezari, 2010). Muraliraja *et al.* (2019) reported that the addition of a C14-SB-type zwitterionic surfactant to the electrolyte resulted in a uniform distribution of nanoparticles in the deposit, a controlled reaction rate, and improved electrolyte clarity, with no decomposition of electrolyte even after 4 h of coating (Muraliraja *et al.* 2019; Gajendiran *et al.* 2025). From the literature survey, it has been observed that nano-sized SnO₂ and Mn₂O₃ exhibit superior properties for improving corrosion resistance in marine environments. However, this specific study has not been conducted in earlier research. Incorporating nano-level SnO₂ and Mn₂O₃ into the binary Ni–P electroless coating results in a composite coating developed on the AH36 steel. It enhances AH36 material with superior corrosion resistance, increased hardness, durability against wear, and potential magnetic/electrical properties. Thus, in this study, Ni–P–SnO₂ and Ni–P–Mn₂O₃ composite coatings were developed on the AH36 steel, and evaluations were made to check their suitability for marine applications. Also, the heat treatment at 400 °C for 1 h was selected to promote Ni₃P phase formation and structural crystallization, which enhances the hardness and corrosion resistance of the Ni–P composite coating. This temperature is widely reported as optimal for improving coating performance without causing oxidation or substrate degradation.

In this work, SnO₂ and Mn₂O₃ nanocrystalline particles were synthesized via a precipitation method and, for the first time, incorporated into an electroless Ni–P matrix for coating AH36 marine steel using a zwitterionic surfactant-assisted process. The optical microscopy analysis was carried out to observe the porosity in the masked surface of the sample. The surface morphology and elements on the deposit were studied using SEM and EDS. The structure and grain size were analyzed using X'pert High Score software. AFM

techniques allow for a thorough analysis of the surface topography. Meanwhile, EDS aids in identifying the elemental composition. The scratch test was employed to evaluate the hardness and resistance of a material to scratches. Corrosion testing was also conducted to determine its ability to withstand environmental degradation. In the Vickers hardness test, a diamond indenter in the shape of a square-based pyramid with a 136° angle between opposite faces is pressed into the material. These techniques provide a comprehensive understanding of the structural, elemental, and corrosion-resistant properties of electroless-coated AH36 steel, demonstrating its effectiveness in maritime applications.

2. EXPERIMENTAL DETAILS

AH36 was selected as the material for electroless coating, with dimensions of 99 × 19.06 × 4.47 mm. A wire electrical discharge machining (EDM) process was used to cut the steel bars to the specified dimensions of 10 × 19.06 × 2 mm. After specimen preparation, the remaining material was retained for future research.

2.1 Preparation of Electrolyte for a Coating Process

Table 1 presents the chemicals used to prepare the electrolyte for a 1 L solution. These ingredients were mixed thoroughly in 150 mL of distilled water. Subsequently, a surfactant solution was prepared to enhance material adhesion and prevent sticking to the coating beaker. For this purpose, 50 mL of distilled water was taken, and a zwitterionic surfactant was added at its critical micelle concentration (CMC); the mixture was stirred until foam formed. For the composite coatings, the electrolyte was prepared for 100 mL. Nanoparticles were dispersed in a separate beaker containing 50 mL of deionized (DI) water and stirred for 30 min before being added to the electrolyte. This surfactant is crucial for promoting better material adhesion and preventing material from sticking to the coating beaker. Once the reaction had started, three drops of the prepared surfactant solution were added.

Table 1. Chemicals used to prepare the electrolyte

S. No.	Chemical	Quantity
1	Nickel sulphate (Source of Ni)	30 g/L
2	Sodium hypophosphite (Reducing agent)	40 g/L
3	Trisodium citrate (Stabilizer)	25 g/L
4	Ammonium chloride (Complexing agent)	100 g/L
5	pH	8–9
6	Temperature	85 °C

2.2 Pretreatment Process

For the pre-treatment process, the sample is thoroughly cleaned with acetone and washed in distilled water. Subsequently, the sample is immersed in ethanol for 2 min, followed by another wash with distilled water. Afterward, the sample is immersed in sulfuric acid, which is prepared in a ratio of 1 mL of sulfuric acid to 9 mL of distilled water. The treated sample is then added to a medium preheated to 85 °C and carefully maintained at a pH of 8–9.

2.3 Electroless Composite Coating on the Substrate

Indirect heating of the electrolyte was achieved using an engine oil beaker kept over the electrically heated magnetic stirrer to achieve the desired temperature in the electrolyte. The electrolyte temperature was monitored using a thermometer every 5 min. The coating process spanned 1 h, and the initial plating bath volume was maintained at 150 mL. Throughout the process, the exposed surface area of the substrate and the electrolyte surface area were held constant. Also, the electrolyte was continuously stirred to avoid the deposition of added nanoparticles at the bottom of the beaker. SnO₂ and Mn₂O₃ nanoparticles were prepared using a precipitation method. Gnanam and Rajendran (2014) provided the procedural parameters for the synthesis and physical characterization of the nanoparticles (Balaraju *et al.* 2016; Zeller and Salvati, 1994). The composition of the coated samples is presented in Table 2.

Table 2. Compositions and processing conditions of Ni–P-based alloy and composite coatings

S. No.	Description	Composition	Condition
S1	Sample 1	Ni–P	Alloy coating
S2	Sample 2	Ni–P–SnO ₂	Composite coating
S3	Sample 3	Ni–P–Mn ₂ O ₃	Composite coating
S4	Sample 4	Ni–P–SnO ₂	Heat-treated Composite coating
S5	Sample 5	Ni–P–Mn ₂ O ₃	Heat-treated composite coating

2.4 Testing of Coated Samples

After the coating, the thickness was calculated using the weight gain method using the following equation:

$$T = \frac{w \times 10^4}{\rho \times A \times t} \quad \dots (1)$$

where, T is the coating thickness, W is the weight difference (after-before weight), ρ is the density, A is the surface area of the substrate, and t is the time

duration of coating. Optical microscopes are fundamental tools in the realm of microscopy, designed to magnify and visualize objects at the microscopic level. The ZEISS Primotech optical microscope with magnification rates set at 50 ×, 200 ×, and 500 × was used to take the images of substrates. Rigaku XRD machines are advanced analytical instruments for material characterization and structural analysis. This machine is within the angular range of 0° to 90° for 2θ measurements. Atomic force microscopy (AFM) is a microscopy and surface analysis technique used for nanoscale imaging and surface characterization. It measures the average surface roughness (R_a) and enables comparison with other samples. The Vickers hardness tester is used to measure the hardness of materials. The applied load on the samples was 200 g for 15 s, and the diagonal lengths of the square-shaped indentation were deduced using an optical system or a microscope. The average of the two diagonals was computed. The Vickers hardness number (HV) is calculated using the formula:

$$HV = \frac{1.854 \times P}{d^2} \quad \dots (2)$$

where, ‘ P ’ is the applied load in kgf, and ‘ d ’ is the average diagonal length of the indentation in mm. Finally, to enhance the properties of the deposit further, selected samples were chosen to undergo an annealing process at a temperature of 400 °C for 1 h. A CH Instruments CHI6112E electrochemical analyzer was used to study the electrochemical behavior of the substrate. The workpiece (AH36 marine-grade steel) with an exposed area of 1 cm² was exposed and used as the working electrode, and it was immersed in a 3.5 wt.% (0.6 N) NaCl solution without deaeration. Platinum wire of size 1 mm was used as the counter electrode, and silver/silver chloride (Ag/AgCl) was used as the reference electrode. To assess the impedance, a frequency response analyser was used in the range from 0.001 kHz to 100 kHz. The scanning rate was 0.1 V/s over the potential range of –1.35 to 0.7 V with respect to the open-circuit potential (OCP). The impedance data are presented as Nyquist plots, representing the average of three measurements for each sample. The upper and lower potential limits for linear sweep voltammetry were set from –1.35 to 0.7 V relative to the OCP after a stable open-circuit potential was achieved. These electrochemical measurements were followed by Tafel plot analysis.

3. RESULTS AND DISCUSSION

The coating thickness of the samples was measured and depicted using the weight gain method. Muraliraja *et al.* (2019) obtained 98.3 μm coating thickness, which was the highest reported in alloy coating. By following the same procedure, in this study, the highest coating thickness of 26 μm was obtained for the composite coating per hour. This could be achieved by introducing the zwitterionic surfactant in the coating

process. The added surfactant would significantly reduce the electrolyte's wetting property. Adding an ammonia solution to maintain pH during the coating process should be the main reason for the increase in deposit thickness. The hardness of the deposits was measured, and the indentations are shown in Fig. 1. The figure depicts clear indentations for samples 2 and 4. However, the sample with more hardness (sample 3) shows less indentation depth. This indicates that sample 3 has a higher microhardness value than the other samples. A microhardness value of 545 HV_{0.2} was obtained for the composite coating reinforced with Mn₂O₃. Heat treatment of the Mn₂O₃-reinforced sample did not significantly affect the hardness value (380 HV_{0.2}; Fig. 2). In sample 1, the Ni content was 85%, which may account for its greater hardness compared to the composite coatings. In terms of microhardness, the untreated Ni-P-Mn₂O₃ coating showed a significant improvement in hardness.

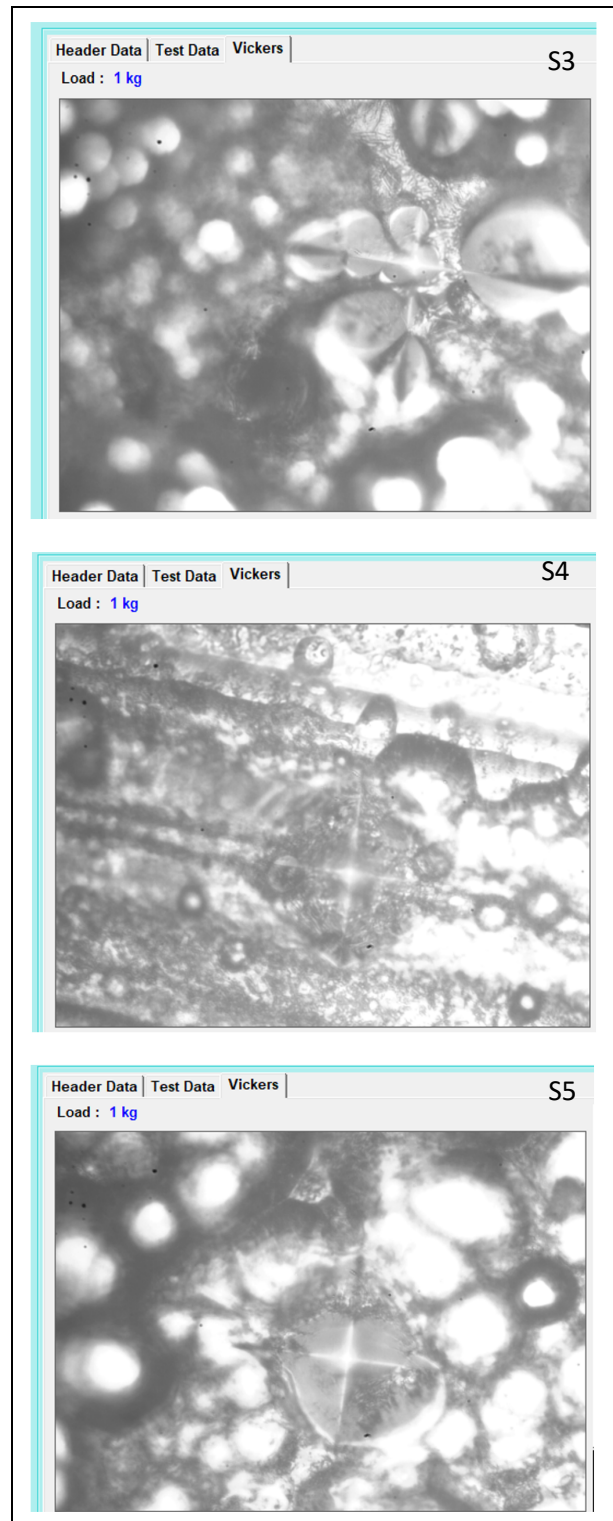
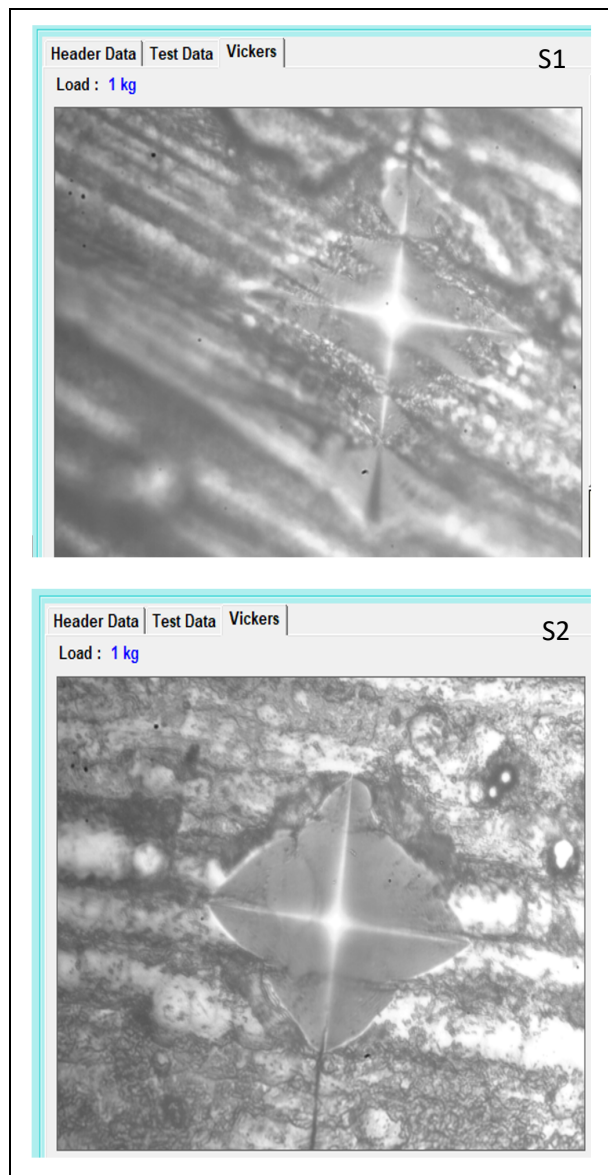


Fig. 1: Indentation of the samples

Incorporation of Mn₂O₃ resulted in an average and maximum microhardness value of 545 HV. Optical micrographs were obtained at a magnification of 200× to compare the morphology with other samples. The image contrast enables clear identification of the heat-treated samples (4 and 5). The grains in samples 4 and 5 appear

more crystalline than those in the other samples (Fig. 3). No visible porosity was observed in the coatings. Thus, the electroless coating was successfully applied to the surface of AH36 steel, providing protection against harsh environments.

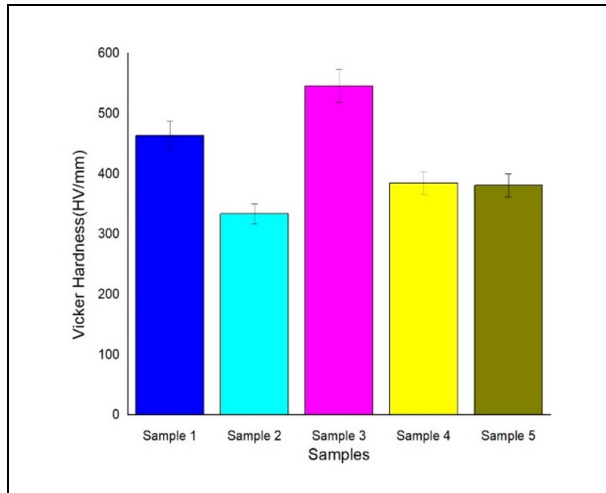


Fig. 2: Microhardness values of the developed samples

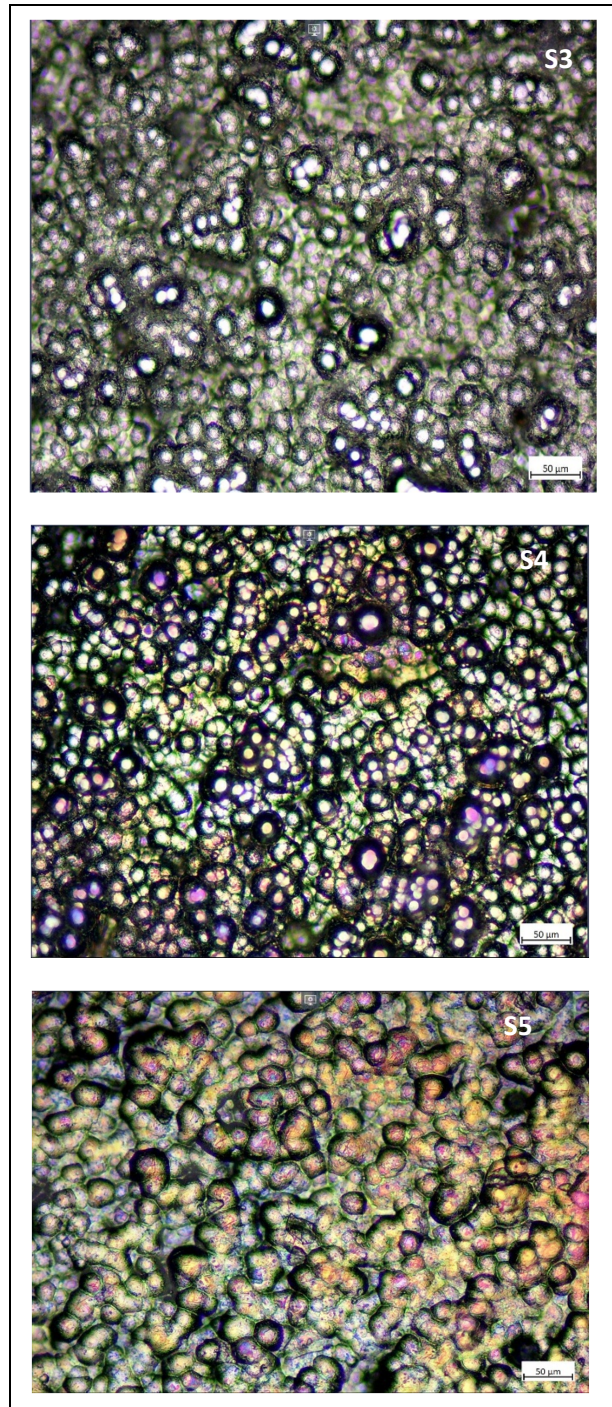
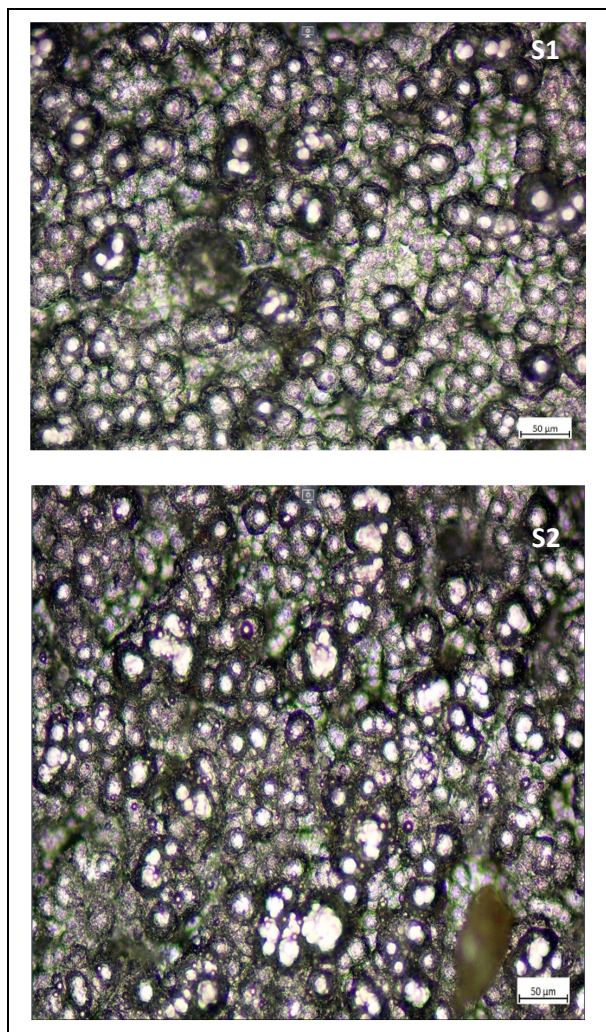


Fig. 3: Optical micrographs of the developed coatings

SEM and EDS analyses are made on the coatings and shown in Figs. 4 and 5. In Fig. 4, the regular cauliflower-shaped grains are observed in the SEM images. The grains are equally distributed in even sizes. Thus, grain size/shape is not responsible for enhancing the properties. It must be purely based on the incorporated nanoparticles in the system. The air-cooled heat treatment process is adopted to elevate the corrosion resistance property. From the EDS analysis, the elements are mapped and identified in percentage depicted in Fig.

5, and the percentage of the elements is given in Table 3. From the earlier literature, it was evident that improvement in hardness and wear resistance of electroless Ni-P coatings depends on the P content and the extent of formation of nickel phosphides during heat-treatment. Similarly, when P% increases, the deposit's brightness and corrosion resistance increase (Gnanam and Rajendran, 2014; Balaraju *et al.* 2001). This is why sample 1 got a higher hardness value. Samples 1 and 4 exhibited higher Ni content. However, sample 3 gives a higher hardness value due to the uniform distribution of approximately 3% hard particles throughout the coating.

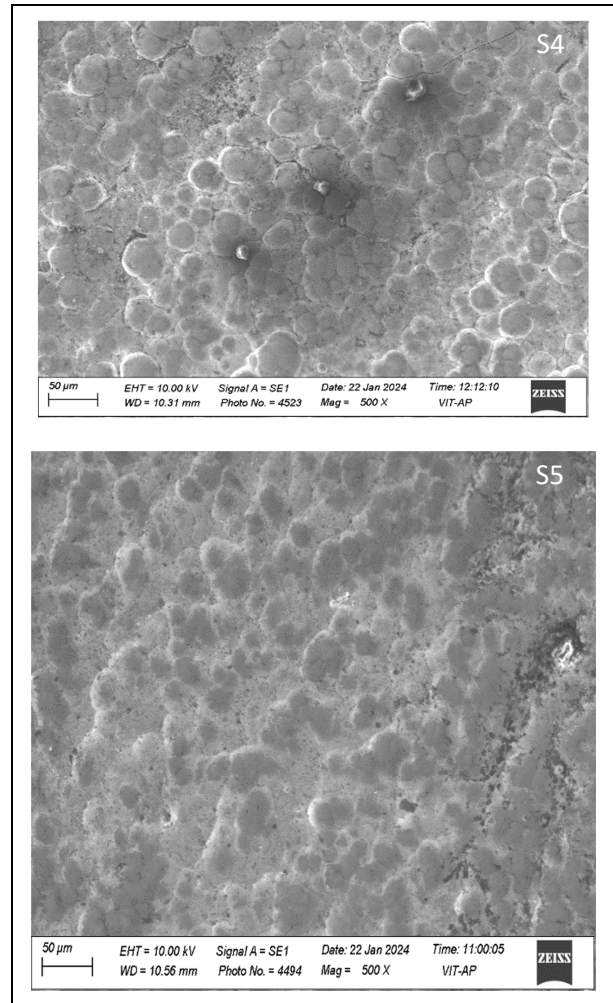
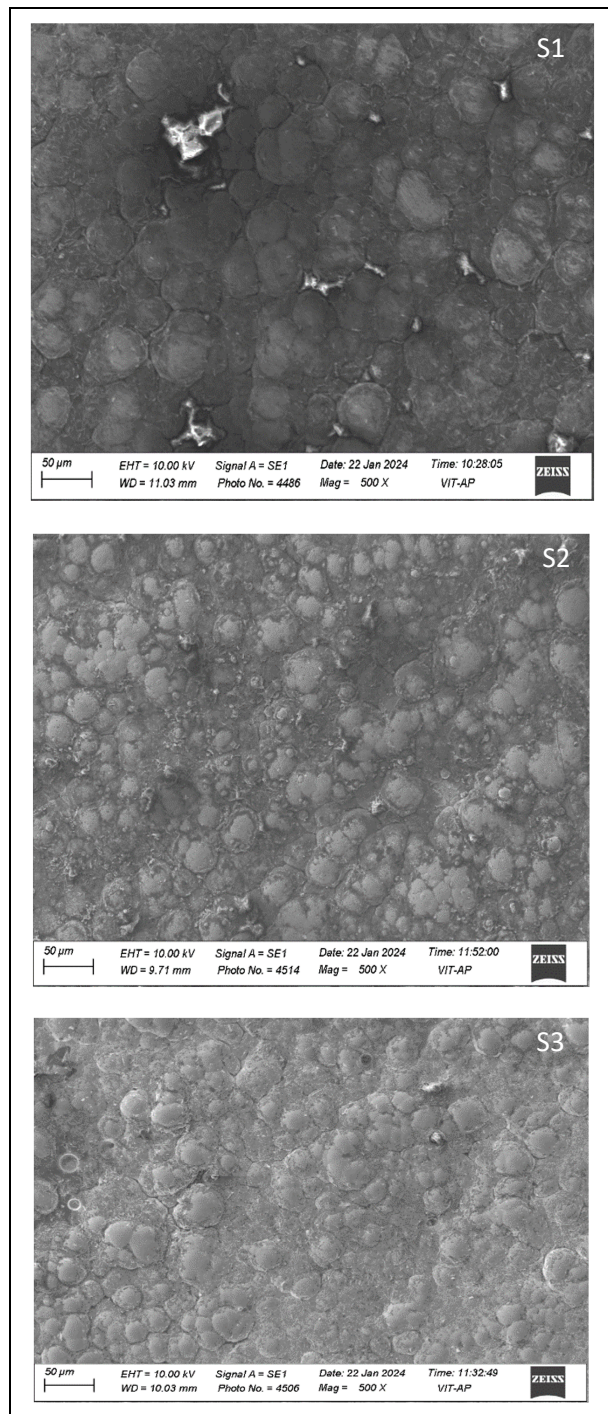


Fig. 4: SEM images of the developed coatings

Table 3. The percentage of elements identified in the deposits

Sample	Ni%	P%	Mn ₂ O ₃ /SnO ₂ %
Sample 1	85%	15%	
Sample 2	76%	20%	4%
Sample 3	74%	23%	3%
Sample 4	81%	15%	4%
Sample 5	75%	22%	3%

The R_a values were obtained by measuring a particular area on the deposit. Three readings were obtained, and the average value was taken as the final value and plotted in the graph shown in Fig. 6. Both heat-treated samples 4 and 5 have smooth surfaces compared to the untreated samples. Surface roughness is one of the factors that could influence the corrosion resistance (Balaraju *et al.* 2016). A quantitative comparison of the measured R_a values should be included by explicitly stating the average roughness of each sample (S1–S5) and calculating the percentage variation between untreated and heat-treated coatings. Correlating the numerical R_a reduction with corrosion current density

(I_{corr}) and microhardness values would technically substantiate the relationship between surface refinement, coating densification, and improved electrochemical performance.

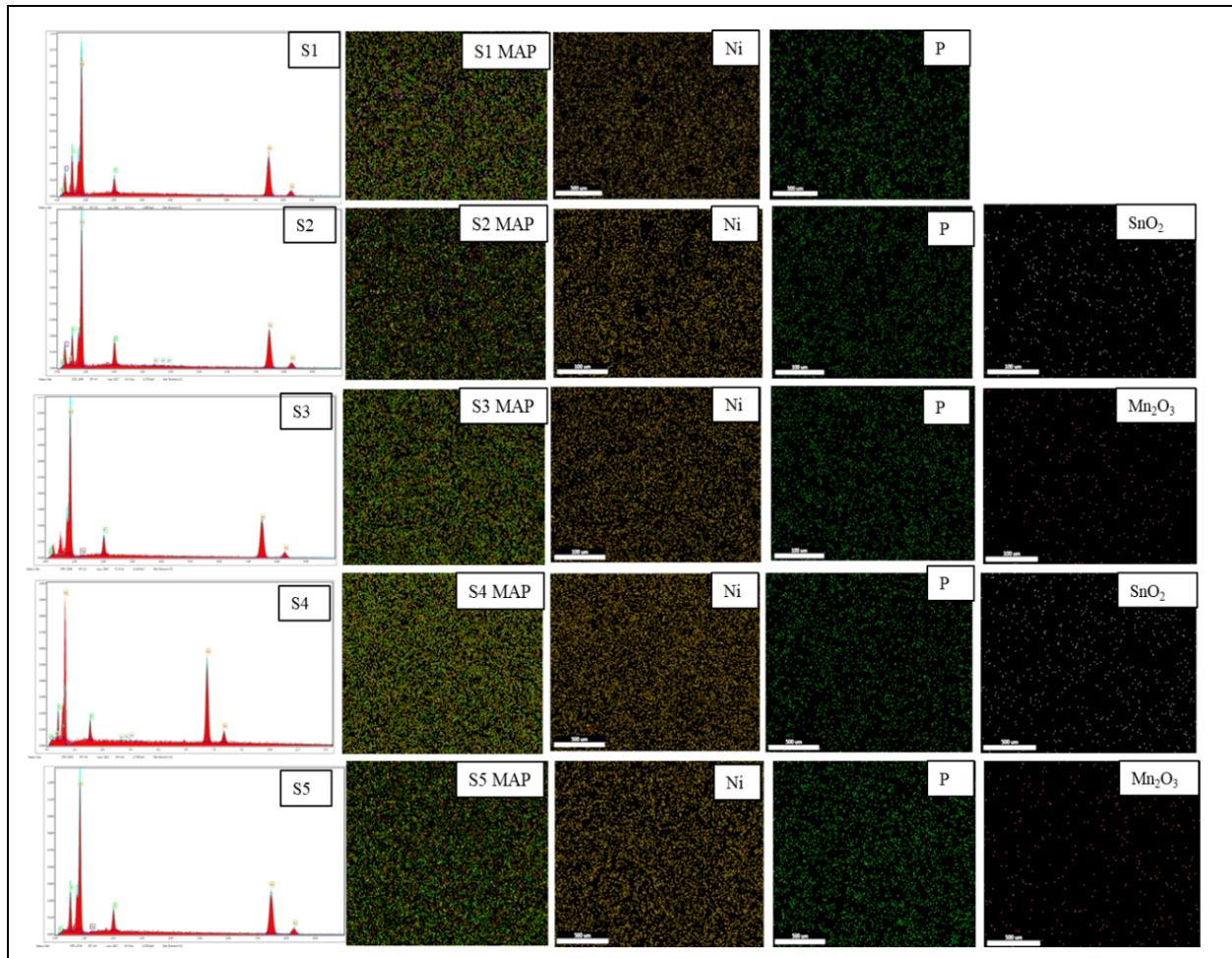


Fig. 5: EDS spectra and elemental mapping of the developed coatings

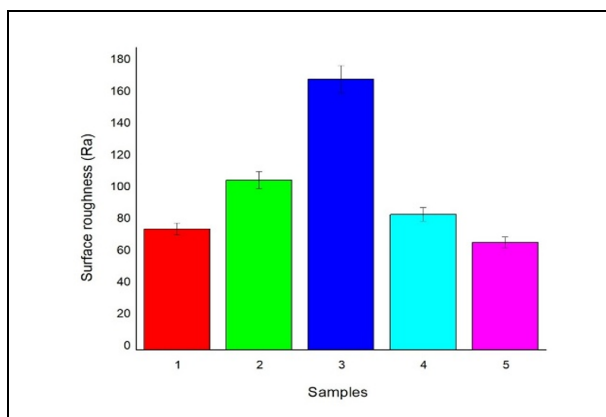


Fig. 6: Surface roughness values of the samples

The XRD diffractogram of the electroless composite coating without and with heat treatment is shown in Fig. 7 (left). The deposits had a single, very broad peak, and the reflections corresponding to the (111) plane of a face-centered cubic nickel phase were

observed for the heat-treated samples 4 and 5. The grain sizes were calculated using the Debye–Scherrer method, and the FWHM and height of the peaks were identified using X'pert Highscore software. The intensity of the (111) peak was much stronger than that of the (200) and (220) peaks in samples 4 and 5. Variations in the full width at half maximum (FWHM), the intensity of the (111) peak, and the nanocrystal size across the samples are shown in Fig. 7 (right side). Heat treatment transforms the amorphous Ni–P matrix into a more crystalline structure with Ni and Ni₃P phases, as confirmed by sharper XRD peaks. Increased crystallinity reduces structural defects, micro-porosity, and active corrosion sites, thereby lowering corrosion current density and enhancing charge transfer resistance in electrochemical measurements. It can be seen that the crystal size decreased with the addition of SnO₂. The peak's maximum grain size and height are observed in the heat-treated Mn₂O₃ sample, which portrays it as highly crystalline. Samples 2 and 3 are not heat-treated substrates; rather, they are composite coatings with a

highly amorphous structure, which contributes to their enhanced corrosion resistance characteristics. It was noted from earlier studies that the crystalline structure helps the material have higher corrosion resistance than the amorphous structure (Zeller and Salvati, 1994).

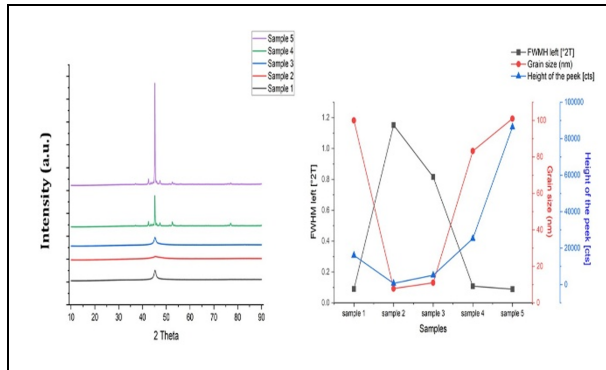


Fig. 7: XRD patterns of the samples (left) and variation of crystallite size, FWHM, and peak intensity derived from XRD analysis (right)

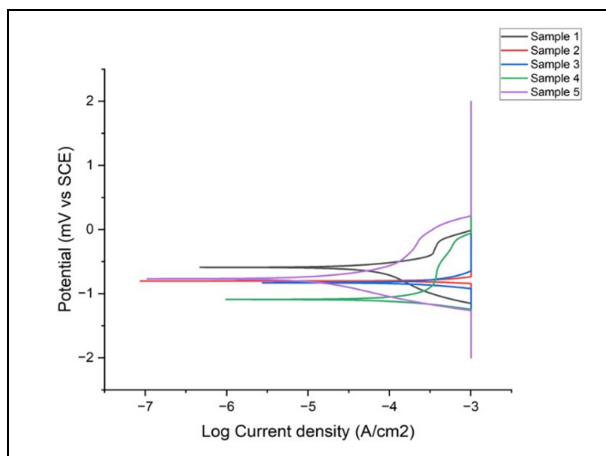


Fig. 8: Tafel plots showing potential vs. log (current density)

Table 4. Tafel parameters of the samples

Sample	Corrosion Potential, E_{corr} (mV)	Corrosion Current Density, I_{corr} ($\mu\text{A}/\text{cm}^2$)
Sample 1	-0.57733	-4.2768
Sample 2	-0.75398	-4.9721
Sample 3	-0.82618	-4.3968
Sample 4	-0.80116	-0.48006
Sample 5	-1.0835	-4.5241

Fig. 8 displays the potentiodynamic polarization curves that were obtained for the electroless plain Ni-P coating as well as the ternary and quaternary alloy coatings as plated in a non-deaerated 3.5 wt.% NaCl solution. The results indicate that the application of composites and the presence of P have enhanced the corrosion behavior. The corrosion potential of the sample

5 electroless coating on mild steel moves to a more positive potential in relation to Ni-P, and the corrosion current lowers. The coating's thickness increases, corrosion current reduces, and corrosion resistance is formed as a result of the nanoparticle increment. As a result, the corrosion current drops to $-0.48006 \mu\text{A}/\text{cm}^2$ as the coating's nanoparticle concentration increases, which is the lowest corrosion current among all evaluated concentrations (Table 4). With increasing nanoparticle content, agglomeration and the rise in solution viscosity reduce the coating thickness and phosphorus content, thereby increasing the corrosion current. The corrosion resistance is increased by the deposition of nanoparticles in the coating, which stops the corrosion cavity from expanding.

The electroless coated substrates were tested for corrosion in 3.5 wt.% NaCl solution at open-circuit potentials. The impedance spectra (Nyquist plots) are shown in Fig. 9 (a–c), recorded over a frequency range from 100 kHz to 0.001 Hz. The plots exhibited a half and a depressed semicircle, suggesting the existence of two relaxation processes within the investigated frequency range. The initial relaxation process is noticeable in the higher and intermediate frequency ranges, while the second relaxation process occurs in the lower frequency range. The depressed semicircles exhibit comparable shapes but vary in diameter. The diameter of each semicircle determines the corrosion resistance of the coatings, with larger diameters corresponding to higher levels of corrosion resistance. By comparing the results, the highest resistance to corrosion is exhibited by sample 5, followed by sample 2, and the rest of the composite coatings, including Ni-P. This could be achieved by adopted air cooled heat treatment process for sample 5. In contrast, in sample 2, the incorporated SnO_2 plays a key role in enhancing corrosion resistance, which is significantly reduced after air-cooled heat treatment (sample 4) (Fig. 9c). The incorporated Mn_2O_3 will significantly enhance the corrosion resistance of Ni-P (Sample 5). Mn_2O_3 performs better than SnO_2 in strengthening primarily due to its stronger interfacial bonding and better load transfer capability within the Ni-P matrix. The Mn_2O_3 particles (3 wt.%) promote dispersion strengthening and grain refinement more effectively, resulting in higher microhardness (545 HV) compared to SnO_2 -reinforced coatings (Anthoni *et al.* 2023; Selvan *et al.* 2022). In real marine environments, coatings are subjected to simultaneous mechanical wear and electrochemical corrosion, leading to synergistic material degradation greater than the sum of individual effects. The incorporation of Mn_2O_3 and SnO_2 nanoparticles into the Ni-P matrix enhances hardness and reduces surface roughness, thereby minimizing mechanical damage, while the dense, crystalline structure formed after heat treatment improves barrier protection against chloride-induced corrosion, making the coating more resistant to tribocorrosion under prolonged marine exposure.

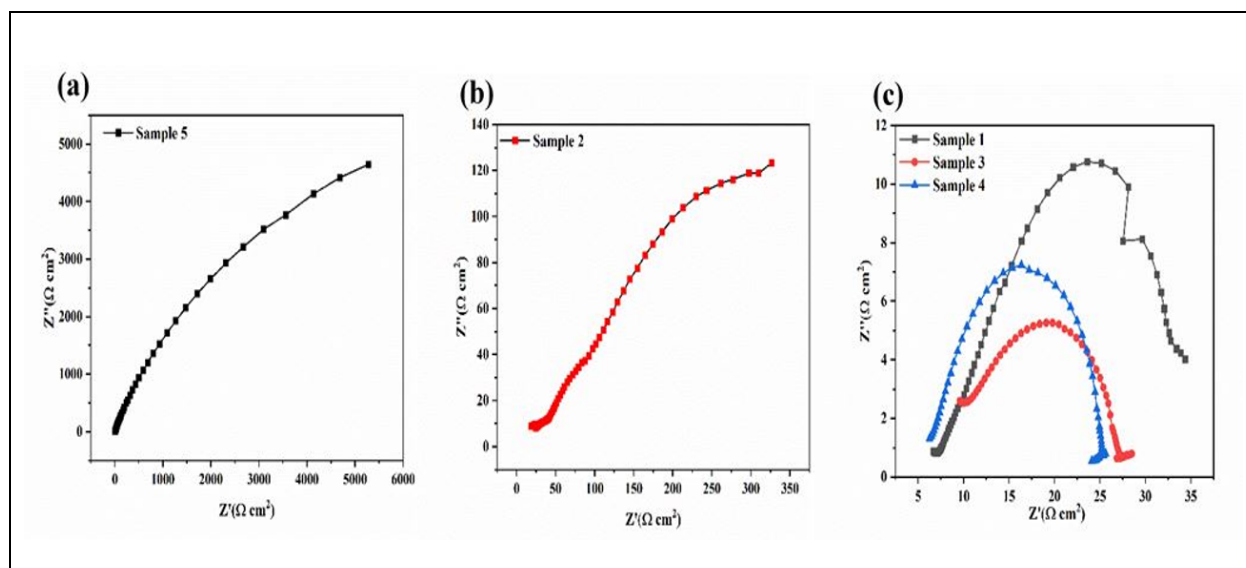


Fig. 9: Nyquist plots obtained from electrochemical impedance spectroscopy (EIS) for Ni-P and composite coatings: (a) Sample 5, (b) Sample 2, and (c) Samples 1, 3, and 4

4. CONCLUSION

Based on the derived results, this study reveals the following key findings:

- The use of a zwitterionic surfactant enhances wettability, thereby increasing coating thickness and achieving uniform deposition on substrates without porosity.
- The addition of synthesized nanoparticles significantly improves the deposit's microhardness value, increasing it from 463 HV (without reinforcement) to 545 HV (with Mn_2O_3 reinforcement).
- Heat treatment reduces the surface roughness of samples, while the strength and corrosion resistance of the deposits are primarily governed by the nickel and phosphorus content.
- Heat-treated substrates exhibit 16.4% superior corrosion resistance compared to the other samples under corrosive environments.

In conclusion, sample 5 (Mn_2O_3 -reinforced, heat-treated substrate) exhibits the highest corrosion resistance among all samples. Nanoparticles with exceptional properties, synthesized via various techniques, offer potential applications that can be further explored using electroless coating processes.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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