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Surface Modification of Cast Iron Rotary Engine Apex Seals Using RoHS-Compliant Electroless Ni–P Coatings

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Abstract

In the present study, Ni-P coatings have been successfully deposited on a difficult to plate cast iron substrates using a RoHS compliant electroless nickel plating bath under optimized pre-treatment conditions. Thick deposits of about 30 µm has been obtained in a plating duration of 4 hrs at 85 °C. Developed coatings were characterized for the surface morphology, composition, structure, adhesion, microhardness and tribological properties. As-plated coatings showed nodular morphology with a composition of 9 wt.% P and balance Ni. Only a single broad peak observed from X-ray diffractrogram obtained for the as-plated coating at 45° 2θ. Indicating the amorphous structure of the developed coating. In order to check the coating adhesion, heat quench test was carried out as per ASTM B571-97. Visual inspection of the coated specimens after the test, observed that there were no blisters formation or coating delamination. This indicates the excellent adhesion between the cast iron substrate and deposited EN coating. Microhardness measurements made on these coatings showed that about 38% increase in hardness values compared to the as-received substrates (350±25 VHN). Roughness measurements (Ra) were made on substrate with and without coating using 3D-Profilometry. These results showed that the developed coatings are marginally smooth (0.16±0.02 µm) compared to the as-received substrates. Reciprocating wear test was carried out on cast iron substrates with and without electroless nickel coating. Reduced wear loss (0.002 mm) and lower coefficient of friction (0.21) values were obtained for coated substrates. An attempt has been made to deposit electroless nickel coating on cast iron rotary engine apex seal part under optimized conditions.

Keywords: Apex Sea, Cast Iron, Coefficient of Friction, Electroless Nickel, RoHS, Wear

1. Introduction

Cast iron is a widely used material, favored for its castability and low cost for manufacturing of complex geometries. These materials exhibit excellent mechanical properties such as tensile strength, toughness, mechanical vibration damping, good fluidity, machinability and wear resistance. Because of the presence of inherent porosity in the cast iron makes it not suitable for strategically important and critical applications. Hence, surface modification of cast iron is specifically required to suit stringent metal finishing processes and also ensuring the reliability for certain manufacturing operations[1]. Among the various surface modification processes, electroless nickel plating is usually preferred coating process due to their uniqueness in achieving uniform coating formation on intricate shapes and difficult to plate metals and non-metallic materials apart from exhibiting excellent corrosion and wear resistance[2,3].

The effect of graphite shape and volume% of graphite presence in several cast iron samples on the strength of electroless Ni-P film has been reported[1].

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It has been concluded that the strength of the plating film was affected by the amount of graphite near the interface between the plating film and substrate and the strength decreased with increasing amount of graphite. It has also been reported that crack initiated at the interface in all cast iron samples (with different vol.% distributed in iron matrix) whereas in the case of mild steel specimen, crack initiated at EN coating film surface based on the crack initiation and propagation monitored change of AE (acoustic emission) count during the bending test.

In a study of electroless nickel plating on SG cast iron, it was observed that the Ni^{2+} reduction is blocked on graphite and leads to a non-covering Ni-P deposit[4]. A pre-treatment by the application of a cathodic electrochemical polarization of the substrate in the chemical nickel bath has proven to be successful to promote a thin homogeneous coating, including on the graphite spheroids[5]. Electroless nickel coating has been developed on ductile or SG iron specifically to improve its corrosion resistance. EIS studies carried out on the substrate and EN coating system found that there is a noticeable increase in the corrosion resistance due to the blocking effect of electroless nickel coating which prevented the access of the electrolyte to the ductile iron[6]. It has been reported that the electroless nickel plating properties of gray cast iron is largely influenced by chemical composition, mechanical properties and surface morphology due to stabilizer concentration and substrate microstructure[7].

In the present study, an attempt has been made to deposit an adherent electroless nickel coating on rotary engine apex seal made of cast iron. Acid electroless nickel bath was used to deposit coating at 85 °C for 4 hrs under optimized pre-treatment conditions. Coated specimens were characterized for their morphology, composition, structure, adhesion, roughness, microhardness and tribological properties. Based on the results, electroless nickel coating has been deposited on cast iron rotary engine apex seals.

2. Experimental

2.1 Sample Preparation

Cast Iron specimens (2.54 cm X 2.54 cm X 0.3 mm) were used as substrates to deposit a uniform and adherent electroless nickel coating. Specimens were cleaned with running water followed by deionized water. Then the specimens were cleaned ultrasonically in acetone for about 8 min. Electro cleaning of these specimens was carried out in mild alkaline solution containing sodium carbonate, sodium hydroxide and tri-sodium phosphate at 75°C, 7 min. anodic and 7 min. cathodic followed by rinsing in running water and deionized water. Further, the substrate was immersed in HCl solution at room temperature for 2 min. with continuous stirring. The specimens were subsequently

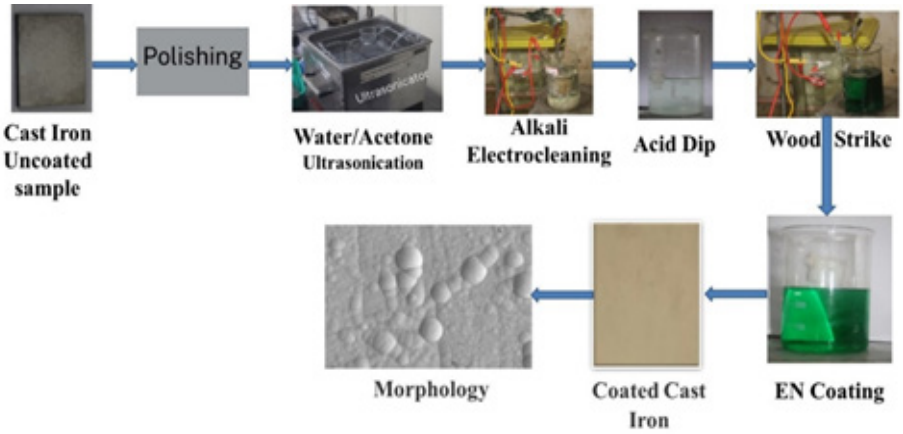


Figure 1. Steps involved in the pre-treatment procedure for cast iron specimens for depositing electroless Ni-P coating.

rinsed with running water followed by deionized (DI) water and subjected to a nickel strike using a Wood's nickel bath. The nickel strike was performed under anodic conditions for 5 min, followed by cathodic deposition for 20 min. A cast iron specimen measuring 1 inch × 1 inch was used as the substrate. A current of approximately 1.3 A was applied, corresponding to a current density of 1.3 A/in^2 (201.5 mA/cm^2). Nickel was used as the anode material. The pictorial representation of the above pre-treatment steps is shown in Fig 1. Then the specimens were immersed in a RoHS compliant proprietary (GIN 625) acidic electroless nickel plating bath (pH 5) which contains lead- and cadmium-free stabilizers, utilizing nickel sulphate and sodium hypophosphite as the nickel source and reducing agent, respectively, to deposit a Ni-P coating. The electroless Ni solution contained in a 500 ml glass beaker was kept in a constant temperature water bath (Siskin Julabo VPC model) at 84±3 °C. All the specimens were plated for 4 hrs with a deposition rate of ~ 7 µm/hr. After plating, the samples were again rinsed in running water and DI water and dried. These specimens were baked at 190^0 C for 3 hrs and used for further characterization.

2.2 SEM, XRD, AES and Optical Studies

Field Emission Scanning Electron Microscopy (FESEM, Supra 40 VP (Carl Zeiss)) was used to analyse the surface morphology, SEM with EDX (energy dispersive X-ray analysis, Oxford Instruments Analytical, UK) attachment was used to determine the elemental composition of the deposits. The bulk chemical composition of the cast iron substrate was analyzed using Arc Emission Spectrometry (AES; Baird, USA, Model: DV6). The measurements were carried out in accordance with the test method specified in DD ENV 12908:1998. X-ray Diffraction (XRD) measurements of the deposit were made in as-plated conditions with a Rigaku D/mx 2200 powder diffractometer using Cu K_α radiation. Cross-section of the deposits were analysed by using an optical microscope (Olympus SZ61).

2.3 Adhesion Test

The adhesion properties of the EN coatings were assessed using a heat quench test as per ASTM B571 – 97. The coated cast iron specimens were heated to a temperature of 250°C and maintained at this temperature for 1 hour. Subsequently, the samples were rapidly quenched in cold water to evaluate the adhesion characteristics of the coating.

2.4 Roughness

The surface morphology and roughness of the samples were characterized using a 3D Stylus Profilometer (Dektak Pro). Measurements were carried out on both the as-received cast iron substrate and the electroless nickel-coated specimens to evaluate the changes in surface roughness resulting from the coating process.

2.5 Microhardness

Microhardness measurements were made on both as-plated and heat treated electroless nickel coatings. Heat treatment was carried out on some of the specimens and were heat treated for 1 hour at a temperature of 400 °C in a muffle furnace. To prevent the oxidation of the coating, a paste of proprietary mixture was uniformly applied on the coated specimens, dried and wrapped in a nickel foil and introduced into the furnace when the required temperature was attained. Heat treatment lasted for 1 hour and the samples were cooled to room temperature in the furnace itself. Wilson Hardness Tester (Buehler make, Model VH1102) was used to measure microhardness of both coating by employing a load of 50gf. Five readings were taken on each deposit and the values were then averaged.

2.6 Tribological Studies

The wear resistance of the samples was assessed using a reciprocating wear tester (Rtec Instruments, Model MFT2000A). The test employed a ϕ6.35 mm hardened steel ball as the counter surface, with a load of 5 N applied on each sample. Each test was conducted for

a duration of 60 minutes, with a stroke length set at 10 mm per second. The total sliding distance for each 60-minute test was calculated to be 60 meters. The wear resistance was evaluated on electroless nickel deposited cast iron specimen (2.54 cm × 2.54 mm × 0.3 mm) in as-plated and heat treated conditions.

3. Results and Discussion

3.1 Morphology and Composition

Morphology of the cast iron substrate is shown in Figure 2(a). From the figure it is evident that the presence of graphite flakes in the iron matrix which is a typical signature of grey cast iron. It is also clearly seen the presence of small pores distributed entire surface of the substrate. Under optimized pre-treatment conditions electroless nickel plating has been carried out for about 4 hrs. From the figure 2(b) it is evident that EN coating exhibited a characteristic nodular morphology. It has been reported that this nodular morphology is due to the nucleation and growth of Ni-P during the plating process apart from substrate roughness[8]. Metallurgical

Table 1. Composition analysis of cast iron substrate and electroless nickel coating

Cast Iron Substrate								
Element (Wt.%)	C	Si	Mn	S	P	Ni	Cr	Mo
	3.01	1.92	0.508	0.058	0.030	---	0.381	0.627
Electroless Nickel Coating								
Element (Wt.%)	Phosphorus				Nickel			
	9.0				91.0			

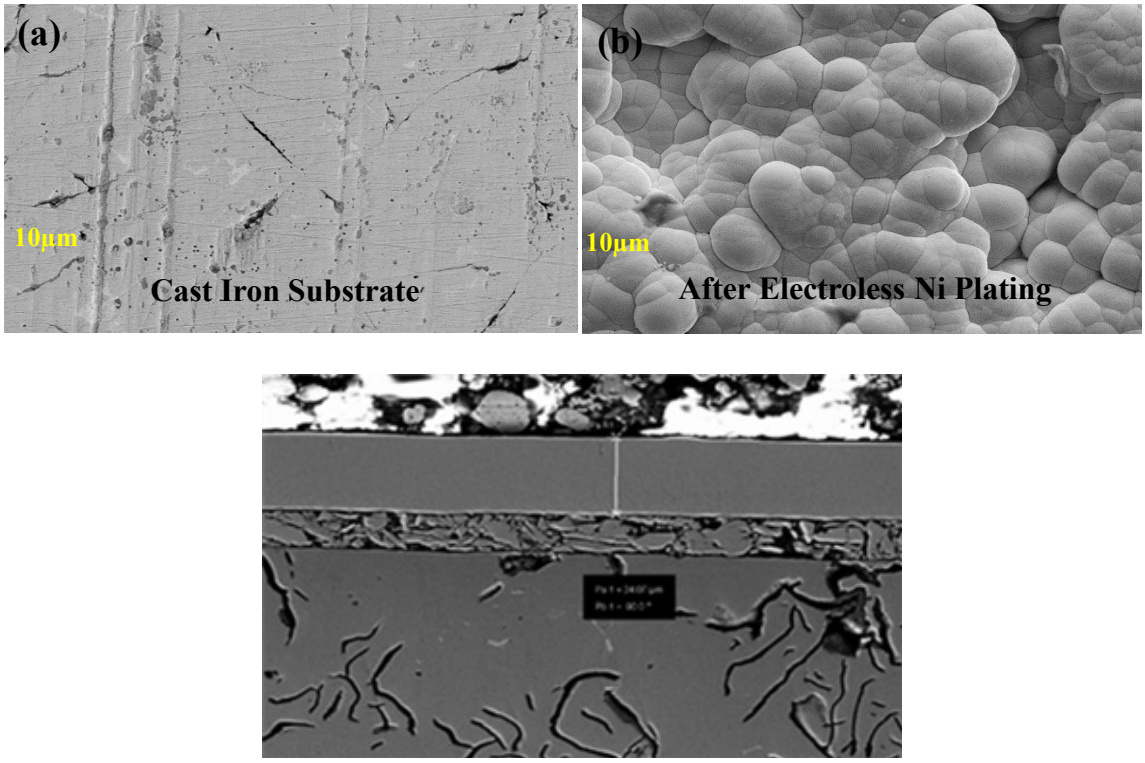


Figure 2. Morphology of electroless nickel coating **(a)** Cast Iron substrate before coating; **(b)** After coating; and **(c)** cross-section of the coating.

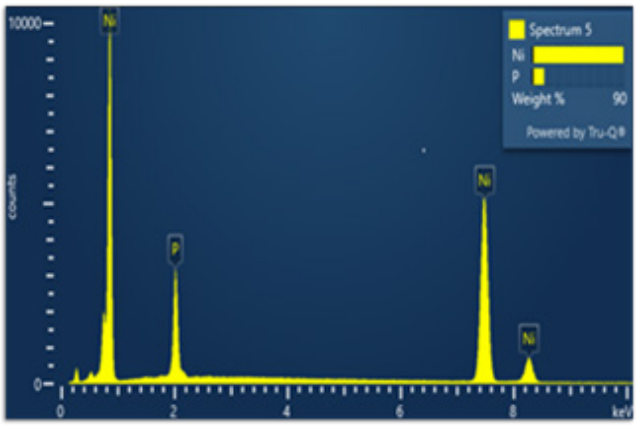


Figure 3. EDX spectra obtained for electroless nickel coated cast iron substrate.

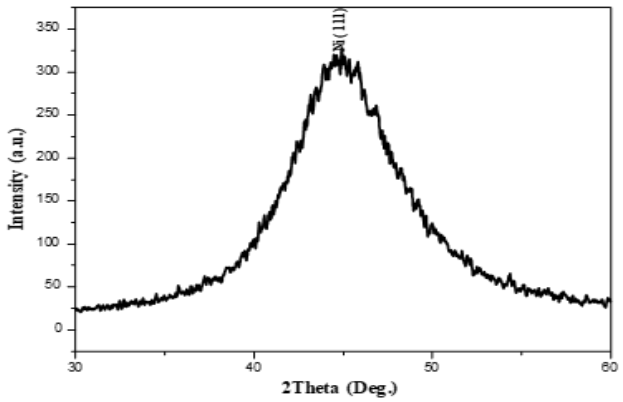


Figure 4. X-ray diffraction pattern obtained for electroless nickel coated cast iron substrate.

cross-section specimen of coated cast iron substrate is shown in Figure 2(c). Coating thickness obtained after 4 hrs of EN plating duration is about 30 µm with a deposition rate of about 7 µm/ hr

Composition of the cast iron substrate and electroless nickel coating have been analysed using optical emission spectroscopy and scanning electron microscopy, respectively. From the table 1 it is evident that substrate is having 3 wt.% of carbon, about 1.9 wt.% of Si apart from other alloying elements (<1 wt.%) such as S, P, Mn, Cr, Mo with a balance Fe. EDX analysis of coated substrate showed about 9 wt.% of P with balance Ni. EDX spectra obtained for EN coating is also seen in figure 3 and the presence of distinct peak corresponding to P apart from Ni peak are clearly seen. It is well known that if the P content is more than 8 wt.% are called high P electroless nickel coatings. There are no peaks pertaining to Fe or C (from substrate) can be seen from EDX spectra since, in the present study, thick EN coatings were deposited on cast iron substrate.

3.2 X-ray diffraction (XRD) analysis

X-ray diffraction pattern obtained for electroless nickel coated cast iron substrate is shown in the Figure 4. A single broad peak can be seen from the figure which corresponds to Ni (111) at 45° 2θ. Theoretically, it has been reported that a disorder in arrangement of atoms manifest itself as a broad peak in X-ray diffractogram[9].

From the composition analysis it is evident that the codeposited P content along with the Ni is high and is about 9 wt.%. It is well known that high phosphorus coatings (> 7 wt.%) exhibit amorphous structure which is evident from the XRD pattern obtained in the present study[10].

3.3 Coating Adhesion

Coating adhesion has been checked as per ASTM B571-97 (heat quench test). From the Figure 5 it is evident that after heat quench test there are no formation of blisters or coating delamination. This indicates the obtained thick electroless nickel coating is intact with the cast



Figure 5. Adhesion testing of electroless nickel coated cast substrate as per ASTM B571-97.

iron substrate. In general, obtaining an adherent coating is difficult particularly challenging to plate substrates like cast iron[2]. Presence of graphite flakes in the Fe matrix makes it difficult in obtaining good adhesion of the developed coating. Hence, pre-treatment plays an important role for obtaining an adherent electroless nickel deposit. In the present study, the optimized pre-treatment resulted in obtained a uniform and adherent coating on difficult to plate substrates like cast iron.

3.4 Coating Roughness

Roughness profiles of cast iron substrate (as-received) and after electroless nickel coating has been measured subsequent to tribology test and the profiles obtained for both are given in Figure 6. One of the important characteristic parameters of the roughness profile is the Ra, is a measure of the arithmetic mean of the departures of profile from the mean line. From the figure it is evident that the Ra value obtained for cast iron is relatively higher (0.19 µm) when compared to the EN coated substrate (0.16 µm). It indicates that after electroless nickel coating the surface has become marginally smoother compared to as-received cast iron substrate.

3.5 Microhardness

Microhardness measurements were carried out on both the uncoated cast iron substrate and the electroless Ni–P coated specimens using a microhardness tester. The optical micrographs of the corresponding indentation impressions are presented in Figure 7. The microhardness values obtained for the substrate and EN coating are 350±25 VHN and 585±15 VHN, respectively. Instrumented indentation testing was performed to evaluate the micro-hardness of the electroless Ni–P coating. Since, in the present study, the Ni-P deposit coating thickness is approximately 30 µm, the substrate influence on the measured hardness

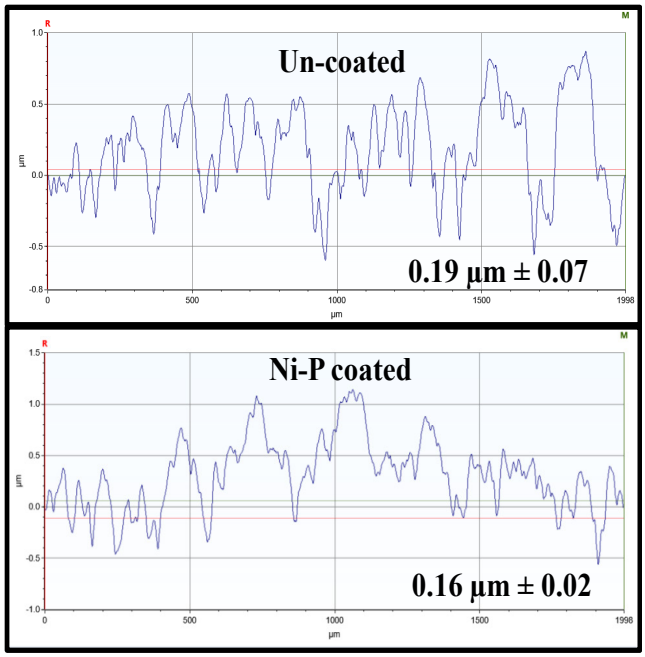


Figure 6. Surface roughness profiles obtained for cast iron substrate before and after electroless nickel coating.

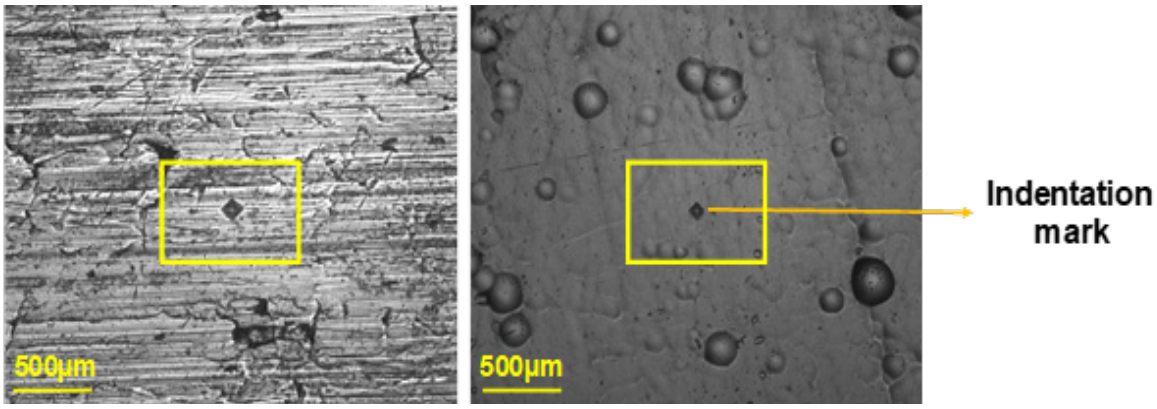


Figure 7. Surface microhardness measurements made on cast iron substrate and electroless nickel coating.

was considered negligible. This is in consistent with the widely accepted recommendation that the maximum indentation depth should be limited to < approx. 10% of the coating thickness to minimize substrate effects during hardness measurements. Therefore, the measured indentation response primarily represents the intrinsic mechanical properties of the coating rather than those of the cast iron substrate[11,12]. It is evident that as-plated electroless nickel coating exhibited about 38% increase in microhardness values compared to the as-received cast iron substrate.

3.6 Tribological Studies

Reciprocating wear test was performed on cast iron substrate with and without coating under cyclic sliding motions. It was observed that the wear loss obtained for the substrate without coating was about 0.005 mm with a Coefficient of Friction (CoF) value of 0.48. In the case of EN coated substrate, the obtained wear loss as about 0.002 mm with CoF value of 0.21. Wear scratches obtained after the test is shown in figure 8. From the figure it is clear that the wear scratch observed on the as-received cast iron substrate was wider and exhibited a dull appearance, indicating significant material removal during the wear test. In contrast, the electroless nickel-coated sample showed a narrower and smoother wear track, demonstrating improved wear resistance and reduced material loss[13]. This is due to the higher microhardness exhibited by electroless nickel coating compared the cast iron substrate. It also depends on the surface roughness and in the present study it is evident that electroless nickel coating exhibited smooth surface compared to the substrate. From this it is clear that electroless nickel coating exhibited better

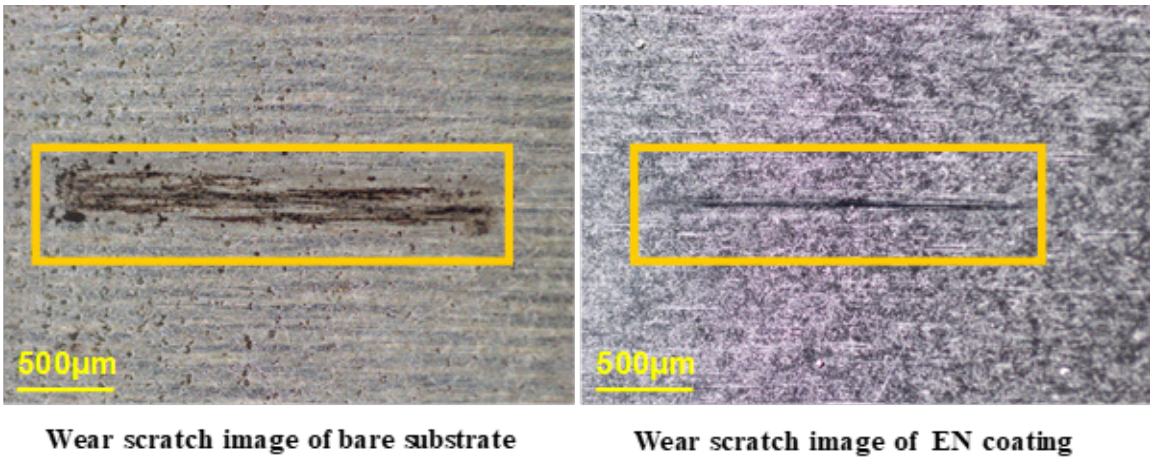


Figure 8. Surface images of cast iron substrate and electroless nickel coating after the wear test.

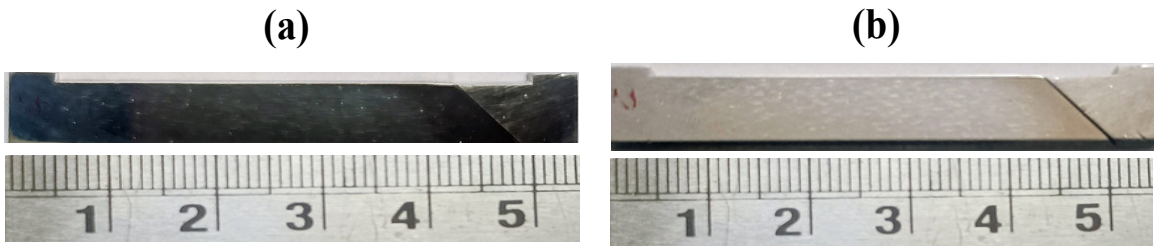


Figure 9. Electroless nickel plating of cast iron rotary engine apex seal (a) as-received; (b) EN coated.

tribological properties compared to the as-received cast iron substrate.

In the present investigation, under optimized pre-treatment conditions, the deposition and characterization of thick electroless nickel coatings were successfully carried out on difficult-to-plate cast iron substrates. These coatings exhibited improved hardness and tribological properties. Based on the above, an attempt has been made to deposit on cast iron rotary engine apex seals. Figure 9 shows the cast iron rotary engine apex seal with and without coating. From the figure it is evident that the silvery appearance of electroless nickel coated apex seal is shiny with smooth surface compared to the as-received cast iron apex seal. Performance evaluation of these coated apex seal is in progress.

4. Conclusions

In the present investigation, high phosphorous thick and uniform electroless nickel coating has been successfully developed on difficult to plate substrate material like cast iron using a RoHS compliant electroless nickel plating bath. Heat quench test carried out on coated cast iron substrate showed that the coating is adherent with the substrate without any blisters or coating delamination. A single broad peak obtained for the high phosphorus electroless nickel coating indicated the amorphous nature of the deposit. Microhardness measurements made on the electroless nickel coating showed about 38% higher microhardness values than the cast iron substrate. Improved tribological properties with lower wear loss and coefficient of friction values obtained for these coatings showed its durability for the practical applications in the manufacturing sector utilizing components made of cast iron.

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Author Contribution

T. Manikandanath: Conceptualization, Methodology, Investigation, Data curation, Formal analysis, and Writing – original draft. K. Sai Vineeth: Experimental investigation, Data collection, and Characterization. J. N. Balaraju: Supervision, Technical guidance, and Review & editing. M. Ganesh: Experimental work. B. Shri Prakash: Data analysis. Sai Venkata Pramod Kumar: Data collection.