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Impact analysis of Electroless Nickel Coating mixed with Alumina Particles

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Abstract: - *Electroless nickel (EN) coating is widely used in industrial applications due to its superior corrosion resistance, hardness, and uniform deposition on complex surfaces. Incorporating alumina ($Al_2 O_3$) particles through co-deposition further enhances the mechanical and wear properties of the coating. This study investigates the impact of electroless nickel coating combined with alumina particle co-deposition on the surface characteristics, mechanical strength, and tribological performance of metallic substrates.*

The research involves the preparation and deposition process using a Ni-P-based electroless bath with varying concentrations of alumina particles. Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) are used to analyze surface morphology and particle distribution, while micro hardness, wear resistance, and corrosion behavior are evaluated through standardized testing methods.

The results indicate that alumina co-deposition significantly increases surface hardness, improves wear resistance, and enhances corrosion protection compared to conventional electroless nickel coatings. Optimal particle concentration and bath parameters are identified to maximize coating performance. This research contributes to the development of advanced composite coatings for applications requiring high durability, improved surface protection, and enhanced functional properties in industries such as automotive, aerospace, and manufacturing.

Keywords –SEM, composite coating, wear, hardness; electroless NiP.

I. INTRODUCTION

To have an environment friendly process for surface coating and to replace the hard chrome surface treatment technique Electroless nickel phosphorous (NiP) coating was developed and was patented [1] in the mid of 20th century. NiP coating is one of the surface treatment technique used in industries due to its hardness, corrosion and wear resistance. Incorporation of second phase particles in the metal matrix lead to enhancement [2] of its physical and mechanical properties. SiC, Al2O3, WC, TiO2, ZrO2, B4C and diamond are the few hard particles co deposited in NiP coating for the hardness enhancement, wear resistance and corrosion resistance. PTFE, MoS2 and graphite are the lubricant solid particles which reduce the friction coefficient and wear rate. Incorporation of micron sized particles of diamond showed improvement in wear resistance of NiP coating [3]. Electroless composite coating uses the conventional reduction reaction process with suspensions of particles. However the successful co-deposition depends on the bath chemical composition particle size and bonding of the particle with metal matrix. Since the granularity of the particle are large, micron particles are not uniformly distributed on the NiP coating and the performance of the coating has not fully achievable. The electro deposition of nickel and co deposited SiC particles [4] increases the wear resistance with uniform distribution of particle in the matrix. In this recent era nano-technology has been developed and nano particle based coating has been received wide attention for its unique properties. On the other hand nano particles have high surface area and possible to get agglomerate. The efficiency of co-deposition of SiC and Si3N4 nano particles [5] depends on particle nature and not based on its size. Analysis on the distribution of nano Al2O3 particle [6] were done and the results showed nano Al2O3 particles are homogenously distributed in nickel using electrolytic co-deposition. The distribution of particles depends on the free powder well isolated and dispersion [7] not depends upon the concentration of particle in the coating bath.

The present work aims to study the surface morphology, chemical composition, hardness and wear resistance of as deposited NiP and Al2O3 particles co-deposited NiP coatings.

II. EXPERIMENTAL DETAILS

Electroless NiP coatings are deposited on the mild steel surface using dip coating technique, where nickel chloride used as source and sodium hypophospite used as reducing agent. Nano Al2O3 particles are mixed in the electrolyte to deposit composite coating. The chemical used for the preparation of electrolyte and the operating conditions [8] of electrolyte were listed in Table 1. Pretreatment of the substrate involves oil and dirt removal from the surface followed by surface activation of substrate. The surface of the substrate were cleaned using soap followed by distilled water wash and cleaned by acetone for 5 minutes. Before starting the coating process in electrolyte solution the surface is activated using 10% HCl solution for 2 minutes. The Al2O3 particles of size 80 nm to 100 nm were ultrasonicated in distilled water prior adding into the electrolyte in order to avoid agglomeration of particles. The ultrasonicated Al2O3 particles were added in the electrolyte and continuous stirring is maintained to have uniform distribution of particles in the coating. The coatings were characterized by X-ray diffraction technique using computer controlled Bragg-Berntano configuration with copper radiation (Cu Kα and λ=1.54Å). The X-ray tube was operated at 30 kV and 40 mA. The surface morphology and elemental chemical composition were analyzed by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) using JEOL JSM-7001F scanning electron microscopes. The hardness of the coatings were experimentally found using load-creep-unload procedure in Dynamic Ultra Micro Hardness Tester type DUH211 (SHIMADZU) equipped with Berkovich (Triangular 115°) indenter. Measurements were recorded for a maximum load of 200 mN and 10 indentations were performed at difference place of the sample for ensuring accuracy. Wear tests were performed using PLINT TE 66 microscale abrasion tester. During the test a directly driven steel ball was rotated against the coating with a load of 2.5 N. The ball rotates with a sliding velocity of 0.2 meter per second in dry condition. The wear scar produced in the specimen surface is spherical geometry of the ball. The wear volume loss (V) can be calculated [9] based on the equation

$V= \pi b4 /(64 R)$ for $b << R$

Where, b is the diameter of the wear crater

R is the diameter of the steel ball

TABLE I ELECTROLYTE BATH CHEMICAL COMPOSITION AND OPERATING CONDITION

Chemical Composition Of Electrolyte		**Concentration (g/l)**	
		NiP coating	***NiP-Al2O3 coating***
Nickel Chloride	$NiCl_2$	40	40
Sodium Hypophosphite	$NaH_2O_2P.H_2O$	30	30
Tri Sodium Citrate	$Na_3C_6H_5O_7$	25	25
Ammonium Chloride	NH_4Cl	50	50
Sodium dodecyl sulfate	$NaC_{12}H_2.5SO_4$	0.6	0.6
Aluminum oxide	Al_2O_3	0	0.1
Operating Condition			
Temperature		87ºC (±1ºC)	
pH		4-5	

III. RESULTS AND DISCUSSIONS

The XRD patterns of as deposited NiP coating and NiP-Al2O3 coatings are shown in Figure 3. A broader and single peak centered on $2\theta = 45°$ shows nickel (111) phase is attributable to the amorphous structure of NiP coating. According to nickel-phosphorous phase diagram nickel and Ni3P are possible phases within the temperature range of 90ºC which is the operating condition for the coating formation. Excess amount of phosphorus of in NiP coating deposits in the grain boundaries of nickel there by prevents the nucleation of nickel phase and has resulted in the formation of metastable amorphous nickel phase with no traces of Ni3P peaks. For the phosphorous content more than 7 wt% the amorphous nature [10] of NiP coatings are observed. In the diffraction pattern of NiP-Al2O3 coating, broad peak is observed between diffraction angle $2\theta = 40\text{-}50^o$ as like NiP coating but with two peaks overlapped. The peak at 44.5^o corresponds to nickel (111) and the peak at 43.3^o corresponds to Al2O3 (113) peak. Small intensity peaks at 25.5^o and 35.1^o represents the presence of Al2O3 in NiP-Al2O3 coating and all Al2O3 peaks are not traceable even though its presence was clearly shown on SEM image. However the broad peak in NiP-Al2O3 coating represents the coating is amorphous which has phosphorous content more than 7% [11].

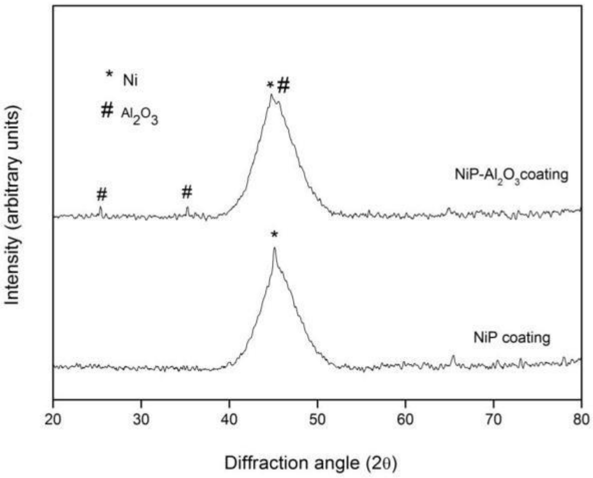
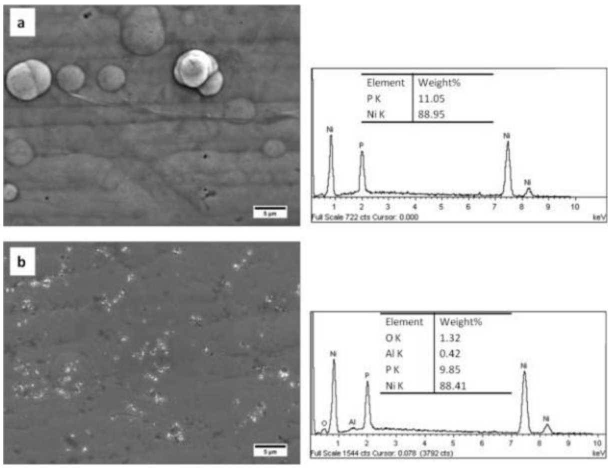


Fig. 1. X-ray diffraction pattern of NiP and NiP-Al_2O_3 coatings



Element	Weight%
P K	11.05
Ni K	88.95

Element	Weight%
O K	1.32
Al K	0.42
P K	9.85
Ni K	88.41

Fig 1. Scanning electron microscope image and EDS (a) NiP coating
(b) NiP-Al_2O_3 coating

Figure 2 shows the surface morphology and EDAX spectrum of NiP and NiP-Al2O3 coatings. The surface morphology of NiP coatings in Figure 2(a) shows a uniform deposition and nodular spherical structure of nickel. The EDS result has been showing the phosphorous content in NiP coating as 11.05%. Most of the researchers agrees that the structure and property of NiP coatings depend on its phosphorous content [12]. A uniform distribution of particles is an important factor in order to obtain better mechanical properties of the composite coating. The surface morphology of NiP-Al2O3 coating is shown in Figure 2(b) where Al2O3 particles are distributed uniformly and embedded in the nickel phosphorous matrix appearing as white dots. The EDS result of NiP-Al2O3 coating shows that reduction of phosphorous content to 9.85% from 11.05%, which indicates the presence of Al2O3 has influence in the formation of crystalline nickel phase.

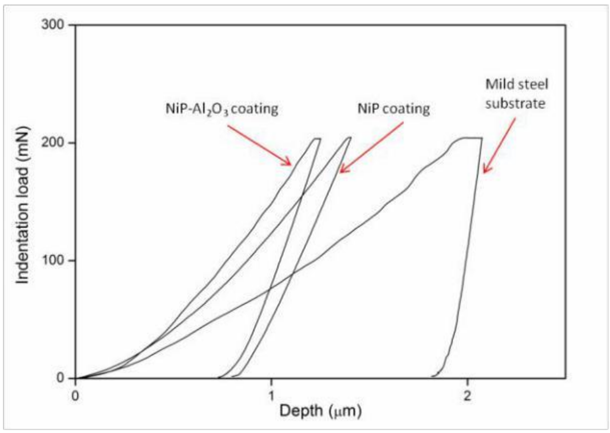


Fig 2. Depth Vs Indentation load on Mild steel substrate, NiP and NiP-Al_2O_3 coatings

Hardness is determined from the ratio of indentation load and contact area. A typical depth – indentation load curve for mild steel substrate, NiP coating and NiP-Al2O3 coatings at the maximum load of 200 mN are shown in Figure 3. Decrease in indentation depth was observed for both NiP and NiP-Al2O3 coating compared with mild steel substrate. The decrease in indentation depth leads to decrease in contact area of the indenter with the coating which in turn increases hardness. The hardness values of the substrate and the coatings were tabulated in Table 2. The co-deposition of Al2O3 particles in NiP coatings results in a minor increase of hardness value from 7.30 GPa to 8.02 GPa. This increase in hardness is due to dispersion strengthening effect by Al2O3 particles.

TABLE II HARDNESS OF SUBSTRATE AND COATING

Type of coating	**Hardness (GPa)**
Mild steel substrate	2.26±0.12
NiP coating	7.30±0.32
NiP-Al2O3 coating	8.02±0.24

The wear behavior in actual service is not as the experimental level, the actual wear volumes will differ from experimental studies but experimentation seems to be speedy and receptive method to know the degradation of the coating. Hence wear scars are produced on the coating with normal load and sliding velocity of rotating ball to be in constant as 2.5N and 0.2 m/s with varying sliding distance. The optical images of the wear scars on NiP coating and NiP-Al2O3 coating for sliding distance of 800 m is shown in Figure 4. It is clearly observed that in NiP-Al2O3 coating the diameter of the wear crater decreases comparerd to NiP coating.

Fig 3. Optical image of wear crater for 800 m sliding distance (a) NiP coating (b) NiP-Al_2O_3 coating

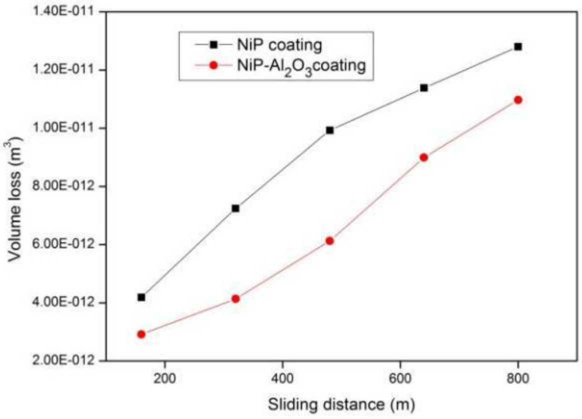


Fig 4. Volume loss Vs sliding distance under 2.5N load, 0.2 m/s sliding speed

Figure 6 shows the volume loss of coating for different sliding distance. Increase in volume loss observed in both NiP and NiP-Al2O3 coatings. Under dry and room temperature conditions the steel ball rolls over the coating surface which leads material transfer from ball to coating and coating to ball. Part of transferred material removed as wear debris and some wear particles are pressed by normal load on the wear crater, then they melt and form oxide films. Metals have high affinity to oxide films and diffusion of metal in oxide film cause strong segregation and prevents direct contact between coating and steel ball. The wear scar of NiP coating has presence of iron oxide over the entire scar and presence of ridge in the circumference of the wear crater which could be due to the accumulation of wear debris formed from the coating and the steel ball. Scanning electron microscope is used to investigate the wear mechanism and the wear crater images shown in Figure 6. From the Figure 6 (a) it is evident that the iron oxide forms a temporary layer over in NiP coating. The formed iron oxide layer has strong influence in the wear resistance of the coating. The absence of micro cuts and grooves on the wear scar of NiP coating shows that the coating is soft enough and is highly adhesive to the substrate. As deposited NiP coating is highly amorphous it is evident that coating is damaged by shear fracture typical of severe adhesive wear.

The presence of Al2O3 particles in the NiP coating decreases wear volume. The co deposition of Al2O3 particles reduces the direct contact of coating by the formation of metal oxide layer and there by reduces the wear rate. NiP-Al2O3 coating shows that the decrease in wear volume is due to presence of metal oxide in the form of ridge on the worn surface. The ridge consists of iron oxide and the oxide layer which is highly adherent to coating that thus protect the coating from further wear. Wear scare SEM image of NiP-Al2O3 coating in Figure 6

(b) shows that presence of nano Al2O3 particles result in wear by abrasion and ploughing of coating. The slackly bound debris is removed away during the ball rotation while strappingly bound particles get entrained between the ball and coating thus creating sharp and deep groves. Thus a combined mechanism of adhesive wear and abrasive wear observed in due to the co-deposition of nano Al2O3 particles in NiP coating and the same mechanism [13] is observed by addition of WC nano particles.

IV. CONCLUSIONS

NiP coating and NiP-Al2O3 coating were prepared using electroless deposition technique. The coating was characterized by X-ray diffraction and Scanning electron microscopy. Hardness and wear tests were performed on the coating and following conclusions are obtained.

1. Both coatings NiP and NiP-Al2O3 are in amorphous phase in as deposited conditions.

2. Al2O3 particles are uniformly distributed during the co-deposition process and has good adherence in NiP matrix.

3. Increase in hardness of coating with due to the co-deposition of Al2O3 particles from 7.30 GPa to 8.02 GPa (increase in 9.8%).

4. Decrease in wear volume loss in NiP-Al2O3 coating compared to NiP coating.

5. Wear crater morphologies shows that adhesive wear observed in NiP coating where as combined adhesive and abrasive wear in NiP-Al2O3 coating.

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REFERENCES

[1] A. Brenner and G. E. Riddell, "Nickel plating on steel by chemical reduction," J. Res. Natl. Bur. Stand.(US), vol. 37, p. 31, 1946.

[2] I. A. Ibrahim, F. A. Mohamed, and E. J. Lavernia, "Particulate reinforced metal matrix composites — a review," Journal of Materials Science, vol. 26, pp. 1137-1156, 1991/01/01 1991.

[3] V. V. N. Reddy, B. Ramamoorthy, and P. K. Nair, "A study on the wear resistance of electroless Ni–P/Diamond composite coatings," Wear, vol. 239, pp. 111-116, 2000.

[4] E. Aghaie, A. Najafi, H. Maleki-Ghaleh, and H. Mohebi, "Effect of SiC concentration in electrolyte on Ni-SiC composite coating properties," Surface Engineering, vol. 29, pp. 177-182, 2013.

[5] M. Sarret, C. Müller, and A. Amell, "Electroless NiP micro-and nano-composite coatings," Surface and Coatings Technology, vol. 201, pp. 389-395, 2006.

[6] B. Müller and H. Ferkel, "Al2O3-nanoparticle distribution in plated nickel composite films," Nanostructured Materials, vol. 10, pp. 1285-1288, 12// 1998.

[7] A. Grosjean, M. Rezrazi, and P. Berçot, "Some morphological characteristics of the incorporation of silicon carbide (SiC) particles into electroless nickel deposits," Surface and Coatings Technology, vol. 130, pp. 252-256, 8/21/ 2000.

[8] S. Karthikeyan and B. Ramamoorthy, "Effect of reducing agent and nano Al2O3 particles on the properties of electroless Ni–P coating," Applied Surface Science, vol. 307, pp. 654-660, 7/15/ 2014.

[9] K. L. Rutherford and I. M. Hutchings, "A micro-abrasive wear test, with particular application to coated systems," Surface and Coatings Technology, vol. 79, pp. 231-239, 2// 1996.

[10] P. Sampath Kumar and P. Kesavan Nair, "Studies on crystallization of electroless Ni P deposits," Journal of Materials Processing Technology, vol. 56, pp. 511-520, 1// 1996.

[11] M. Palaniappa and S. K. Seshadri, "Hardness and structural correlation for electroless Ni alloy deposits," Journal of Materials Science, vol. 42, pp. 6600-6606, 2007/08/01 2007.

[12] K. G. Keong, W. Sha, and S. Malinov, "Crystallisation kinetics and phase transformation behaviour of electroless nickel–phosphorus deposits with high phosphorus content," Journal of Alloys and Compounds, vol. 334, pp. 192-199, 2/28/ 2002.

[13] Y. Y. Liu, J. Yu, H. Huang, B. H. Xu, X. L. Liu, Y. Gao, et al., "Synthesis and tribological behavior of electroless Ni–P–WC nanocomposite coatings," Surface and Coatings Technology, vol. 201, pp. 7246-7251, 5/21/ 2007.