*IOSR Journal of Mechanical and Civil Engineering (IOSR-JMCE)*
*e-ISSN: 2278-1684, p-ISSN: 2320-334X*
*PP 34-37*
*www.iosrjournals.org*

# Study of tribological properties of electroless Ni–P–$Al_2O_3$ composite coatings

Prasanna Gadhari[1], Prasanta Sahoo[1]
[1]*Department Mechanical Engineering, Jadavpur University, Kolkata-700032, India*

***ABSTRACT :*** *Electroless plating technique has been applied to prepare Ni–P composite coatings containing $Al_2O_3$ particles on mild steel substrate. Alumina particles with various contents from 5 to 15 g/L in bath are co-deposited within Ni–P deposits. The effect of coating parameters such as nickel sulfate and sodium hypophosphite concentration on surface roughness, hardness, and tribological properties are investigated. Coatings are characterized by SEM for surface morphology and EDX for analyzing elemental composition. Coating with $Al_2O_3$ concentration of 15 g/L, nickel sulphate of 40 g/L, sodium hypophosphite concentration of 20 g/L, and with heat treatment at 400°C shows maximum coefficient of friction of 0.425, minimum wear loss of 0.36 mg, and micro-hardness of 1506 $HV_{0.05.}$*

***Keywords:*** *Electroless Ni–P–$Al_2O_3$ composite coatings, Roughness, Hardness, Wear, Friction.*

## I. INTRODUCTION

The electroless deposition of metallic nickel from aqueous solution in the presence of hypophosphite was first noted as a chemical accident by Wurtz in 1844[1]. Electroless nickel coating, also known as autocatalytic nickel coating, is produced by the catalytic reduction of nickel ions using reducing agent without the use of electric current. Electroless coatings can broadly be classified into four categories viz, pure nickel and black nickel coating, alloy and poly-alloy coatings, composite coatings and electroless nano coatings [2]. Electroless plating has highly consistent coating thickness across all surfaces, including edges and complex interior geometry. The electroless coating has gained popularity due to its ability to produce coating that possesses excellent wear, corrosion and frictional resistance and hardness [3].

Electroless nickel composite coatings are produced by co-deposition of fine inert second phase particles into a metal matrix from an electroless bath. To improve further the mechanical and tribological properties of the Ni–P deposits, second phase particles have been introduced into the Ni–P matrix. The composite coatings can be categorized into two groups: (1) coatings incorporating soft particles like PTFE, $MoS_2$, HBN, Graphite to provide better lubricity, reduced friction coefficient, and improved corrosion resistance to the coated surface and (2) coatings consisting of hard particles, e.g., SiC, WC, $Al_2O_3$, $Si_3N_4$, $TiO_2$, $ZrO_2$, etc. for getting higher hardness, wear, friction, and corrosion resistance[4]. The excellent properties of electroless nickel composite coatings depend on stable dispersion of composite particles otherwise composite coatings would have non-uniformly distributed particulates and numerous defects, owing to the segregation and agglomeration of the composite particles with high surface energy and activity in the electroless plating bath [5]. An appropriate heat treatment at 400°C for 1 hour can increase hardness and tribological properties such as frictional and wear resistance of composite coatings significantly [6].

$Al_2O_3$ is widely used because of its high elastic modulus, strength retention at high temperature, and high wear resistance [7]. The incorporation of second phase particles in electroless Ni–P composite coatings depends on particle impingement on the coating surface and holding time of the particle on the coating surface [8]. Alirezaei et al.[9] have reported that deposition rate, co-deposition particle percent, roughness and hardness of Ni–P–$Al_2O_3$ coatings have been influenced by concentration of alumina in bath, whereas average roughness and hardness increase with particle content. In this study, Ni–P–$Al_2O_3$ composite coating is developed by electroless process by varying the content of nickel sulphate, sodium hypophosphite and effect of alumina content on the surface morphology, roughness, hardness, friction, and wear have been investigated.

## II. EXPERIMENTAL DETAILS

Mild steel (AISI 1040) specimens of size 20 mm × 20 mm × 8 mm are used as substrate material for the deposition of the electrolessNi–P–$Al_2O_3$ composite coating. Square specimens are prepared very carefully for the deposition of the electroless nickel composite coating. Shaping, parting and milling processes are used accordingly for the preparation of the samples. After that samples are subjected to a surface grinding process. The samples are mechanically cleaned of foreign matter and corrosion products. Then the surfaces of the specimens are cleaned using distilled water. After thorough cleaning, the specimens are given a pickling treatment with dilute hydrochloric acid (50%) for 1 min. Subsequently, they are rinsed in distilled water followed by methanol prior to coating. The cleaned samples are activated in palladium chloride at 55°C and placed in the bath for deposition for 3 hours.

The bath composition and operating conditions for electroless Ni–P–$Al_2O_3$ composite coatings are selected after several experiments, and proper ranges of the parameters are chosen accordingly. The three most important parameters are varied and others are kept constant for coating deposition. Table 1 indicates the electroless bath composition and operating conditions used for the deposition electrolessNi–P–$Al_2O_3$ composite coatings.

Table 1 Electroless bath composition and operating conditions.

| Bath Composition | | Operating Conditions | |
|---|---|---|---|
| Nickel Sulphate | 35, 40, 45 g/L | pH | 4.5-5.0 |
| Sodium Hypophosphite | 15, 20, 25 g/L | Temperature | 85±2$^0$C |
| $Al_2O_3$ Powder | 5, 10, 15 g/L | Bath Volume | 250 ml |
| Tri Sodium Citrate | 15 g/L | Stirrer Speed | 150 rpm |
| Sodium Acetate | 5 g/L | Deposition Time | 3 Hours |
| Sodium Dodecyl Sulphate | 0.2 g/L | | |
| Lead Acetate | 2 mg/L | | |

To have better dispersion of second phase alumina particles, the given amount of surfactant SDS (Sodium Dodecyl sulphate) is added to the electroless nickel poly alloy bath. About 50 ml of electroless nickel solution containing required amount of alumina powder is thoroughly mixed using mortar and pestle. Magnetic stirrer (Remi make) is used to get uniform suspension of particles in the solution. At first a Ni–P layer is deposited for 1 hour to prevent the porosity of the coating and then the solution containing $Al_2O_3$ particles is introduced into the same bath for the subsequent 2 hours Ni–P–$Al_2O_3$ co-deposition.

The composite coatings are isothermally heat-treated for 1 hour at 400°C temperature. The heat treatment is carried out in furnace to improve the mechanical properties of $Al_2O_3$ composite coating. Thereafter, the specimens are cooled down in the furnace to minimize the internal stresses. The hardness of $Al_2O_3$ composite coatings is measured using UHL micro hardness tester with a Vickers diamond indenter. Hardness measurement is carried out on composite coating under a 50 g load for 10 seconds dwell time and indentation speed of 50 µm/sec. Average of five measurements is reported as a hardness value.

Wear characteristics of the EN coated specimens are studied under dry, non-lubricated conditions and at ambient temperature of about 30°C and relative humidity of about 85% in a multi tribotester apparatus (TR-25, DUCOM, INDIA) using a plate-on-roller configuration. The $Al_2O_3$ composite coated specimens in horizontal position are pressed against a rotating steel roller (dia. 50 mm × thickness 20 mm and composition conforming to EN8 specification) with hardness of 55 HRc, placed below the plate. In this method, the plate and the roller are arranged in such a way that the rotating roller serves as the counter-face material while the stationary plate serves as the test specimen. A 1:5 ratio loading-lever is used to apply normal load on top specimen. Wear is measured in terms of loss of weight due to friction. The hardness of the coatings is found to be less than that of the bottom roller (55 HRc). Thus, the bottom roller undergoes negligible wear and the measured wear is essentially the wear of the coated specimen. The frictional force is measured by frictional force sensor that uses a beam type load cell of capacity 1000 N. The test is conducted at 60 rpm for 5 min at normal load of 25 N.

For studying the surface and cross section of the deposits, scanning electron microscopy (SEM) (JEOL, JSM -6360, Oxford Instruments, Japan) equipped with EDX analysis has been used to determine the wt % of the alumina, phosphorus, oxygen, and nickel content in the composite coating. The thickness of deposits is also measured in cross-section by SEM. The roughness measurement of the coatings prior to the tribological testing is done using stylus type profilometer, Talysurf (Taylor Hobson, Surtronic $3^+$). The profilometer is set to a cut-off length of 0.8 mm, Gaussian filter and 4 mm evaluation length.

## III. RESULTS AND DISCUSSION

### 3.1 Surface morphology and roughness

As shown in Fig 1A, $Al_2O_3$ particles are uniformly co-deposited within Ni–P matrix. The agglomeration of composite particles can be prevented by using a suitable anionic surfactant such as Sodium

Dodecyl Sulphate (SDS). Surfactant helps the composite particles for better suspension in plating bath as it increases the surface energy of $Al_2O_3$ particles [10]. Energy Dispersive Spectroscopy (EDS) analysis of Ni–P–$Al_2O_3$ composite coating is shown in Fig1B, which indicates 7.52 wt % of $Al_2O_3$ particles, 6.67 wt % of phosphorus. On the basis of phosphorus content the composite coating can be categorized as medium phosphorus electroless composite coating. Due to higher wt % of $Al_2O_3$ particles the phosphorus percentage reduces. Grosjean et al. [11] reports that changes in surface morphology depend on phosphorus content in composite coatings. Thickness of composite layer is approximately 30 microns which confirms that the deposition rate is about 10 µm/hr. The variation of average roughness value with respect to $Al_2O_3$, nickel sulfate, and sodium hypophosphite concentration is calculated with the help of stylus type profilometer. The result shows that in the presence of SDS a smooth surface is obtained in all samples with $R_a$ of less than 1 µm. $R_a$ value increases with increase in $Al_2O_3$ particles, nickel sulfate, and sodium hypophosphite concentration in bath which confirmed that more amount of alumina particles are embedded into the coating during deposition process. Momenzadeh et al. [12] have observed that surface roughness value decreases due to the presence of SDS in plating bath.

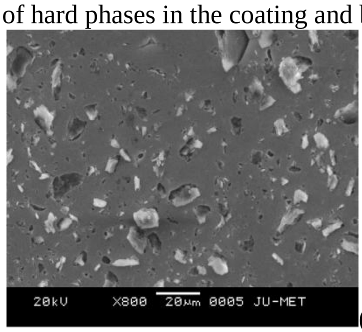
**3.2 Microhardness**

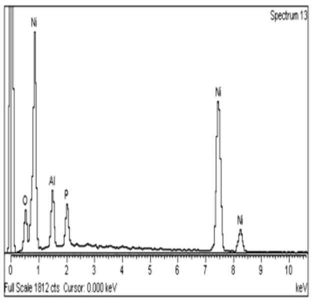
The hardness of electroless Ni−P coating is considerably higher than conventional electroplated nickel and has, therefore, found many industrial applications. Hardness of Ni−P−$Al_2O_3$ composite coating depends on the presence of alumina particles in the coating [9]. Fig 2A shows the variation in microhardness of Ni−P−$Al_2O_3$ composite coating at different concentration of $Al_2O_3$ particles and bath concentration. The result confirms that the microhardness is increases with increase in concentration of $Al_2O_3$ particles in the deposit. The samples prepared by using 15 g/L of $Al_2O_3$ particles, 40 g/L of nickel sulfate and 20 g/L of sodium hypophosphite shows maximum hardness in as-plated condition as well as after heat treatment at 400°C. The hardness decreased at 45 g/L concentration of nickel sulfate and 25 g/L concentration of sodium hypophosphite. After heat treatment, micro hardness of coatings greatly increased due to precipitation of hard $Ni_3P$ particles. A difference of approximately from 50 to 70 $HV_{0.05}$ in hardness is seen within composite coatings in as-plated and from 100 to 150 $HV_{0.05}$ in heat-treated conditions. The higher hardness obtained for composite coatings is due to double strengthening effect from the dispersion strengthening of hard phase and precipitation strengthening of Ni–P alloy. It shows that micro-hardness of annealed composite coatings depends on three factors level of incorporation of particles, annealing temperature and uniform distribution with less agglomeration of particles.

**3.3 Wear and friction resistance**

Wear behavior of Ni–P–$Al_2O_3$ composite coatings at normal load of 25 N, and at speed of 60 rpm is shown in Fig. 3A. The weight loss during the wear test for composite coating having $Al_2O_3$ concentration of 15 g/L, nickel sulfate of 40 g/L and sodium hypophosphite of 20 g/L is lowest under the applied load. Minimum weight loss during test shows that better wear resistance of the said coating compare to other two type composite coatings. In case of Ni–P–$Al_2O_3$ composite coatings incorporation of alumina particles is responsible to increase the wear resistance of the coating. From Fig. 2B it is clear that friction coefficient of $Al_2O_3$ composite coating is vary from 0.225 to 0.425 with increasing amount of alumina particles. Higher values of friction coefficient of composite coatings may be attributed to the incorporation of the hard $Al_2O_3$ particles in the coatings. Composite coating with $Al_2O_3$ concentration of 15 g/L, nickel sulphate of 40 g/L, and sodium hypophosphite with concentration of 20 g/L shows maximum coefficient of friction of 0.425.

From Fig 3B, it is clear that the fewer scratch marks are present on the coating surface. This confirmed that better wear resistance of the composite coating. The wear tracks are mainly characterized by scratches parallel to the relative movement of roller, suggesting that the abrasive wear predominates due to the presence of hard phases in the coating and beneath it [13].



(A)


(B)

Fig. 1. A) SEM image $Al_2O_3$ composite coating, B) EDX of Ni−P−$Al_2O_3$ composite coating.

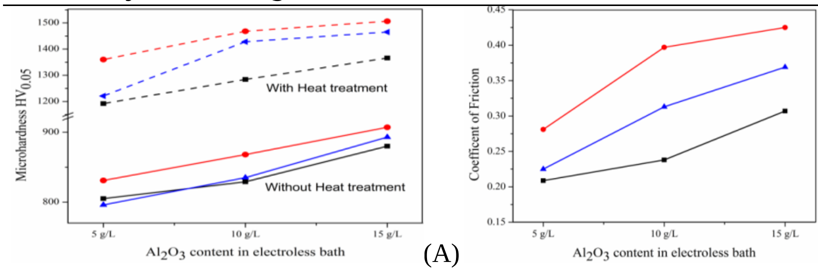



Fig. 2. A) Variation in microhardness of $Al_2O_3$ composite coating, B) Effect of concentration of $Al_2O_3$ particle on friction coefficient.

● - $NiSO_4.6H_2O$ = 40 g/L, $NaH_2PO_2$ = 20 g/L, ◀ - $NiSO_4.6H_2O$ = 35 g/L, $NaH_2PO_2$ = 15 g/L,
■ - $NiSO_4.6H_2O$ = 45 g/L, $NaH_2PO_2$ = 25 g/L

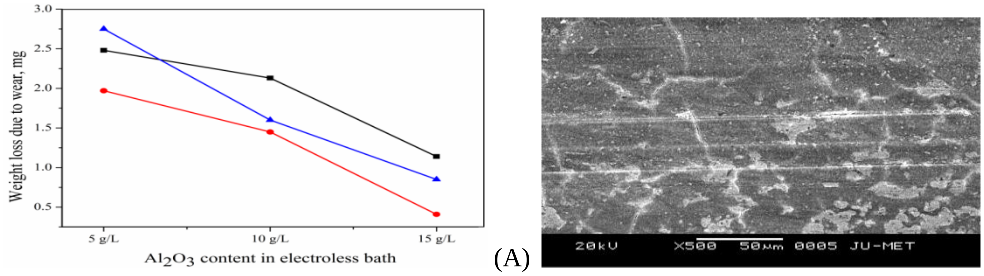



Fig. 3. A) Effect of concentration of $Al_2O_3$ particle on weight loss due to wear, B) SEM image of worn surface of $Al_2O_3$ composite coating.

● - $NiSO_4.6H_2O$ = 40 g/L, $NaH_2PO_2$ = 20 g/L, ◀ - $NiSO_4.6H_2O$ = 35 g/L, $NaH_2PO_2$ = 15 g/L,
■ - $NiSO_4.6H_2O$ = 45 g/L, $NaH_2PO_2$ = 25 g/L

## IV. CONCLUSION

Co-deposition of alumina particles within Ni–P coating matrix changes the roughness and the surface morphology of composite deposits from a smooth state to a non-smooth state with the nodular appearance in Ni–P–$Al_2O_3$ composite coatings. In the presence of surfactant the roughness values vary from 0.3 to 0.6 µm. The hardness, coefficient of friction and wear resistance of $Al_2O_3$ composite coatings increase in the presence of $Al_2O_3$ particles, whereas the maximum hardness and wear resistance are achieved in heat treated coating at about 400°C. Abrasive wear is the most dominant mechanism for wear of Ni–P–$Al_2O_3$ coatings.

## REFERENCES

[1] G. O.Mallory, and B.Hajdu, Electroless Plating: Fundamentals and Applications *Noyes Pubn Bracknell Berkshire* UK 1990.
[2] J Sudagar, J. Lian, and W. Sha, Electroless nickel, alloy, composite and nano coatings-A critical review, *Journal of Alloys and Compounds 571* 2013 183-204.
[3] J. N.Balaraju, T.S.N. Sankara Narayanan and S.K.Seshadri, Electroless Ni–P composite coatings, *Journal of Applied Electrochemistry 33*: 807–816, 2003.
[4] A. Sharma, and A.K. Singh, Electroless Ni–P and Ni–P–$Al_2O_3$ Nanocomposite Coatings and Their Corrosion and Wear Resistance, *Journal of Materials Engineering and Performance*, April 2012.
[5] P. Sahoo, and S. K. Das, Tribology of electroless nickel coating – A review, *Materials and Design*, *32*, 2011, 1760-1775.
[6] I. Apachitei, F.D. Tichelaar, J. Duszczyk, andL. Katgerman, Solid-state reactions in low-phosphorus autocatalytic Ni–P–SiC coatings, *Surface and Coatings Technology 148* 2001 284–295.
[7] A. Abdel Aal, Z.I. Zaki, and Z. A. Hamid, A Novel composite coatings containing (TiC–$Al_2O_3$) powder, *Materials Science and Engineering A 447* 2007 87–94.
[8] J. N.Balaraju, T. S. N. Sankara Narayanan, and S. K. Sheshadri, Structure and phase transformation behaviour of electroless Ni-P composite coatings, *Materials Research Bulletin 41* 2006 847-860.
[9] Sh. Alirezaei, S.M. Monirvaghefi, M. Salehi, and A. Saatchi, Effect of alumina content on surface morphology and hardness of Ni–P–$Al_2O_3$(α)electroless composite coatings, *Surface and Coatings Technology 184* 2004 170–175.
[10] S.A. Abdel. Gawad, A.M. Baraka, M.S. Morsi and M.S. Ali Eltoum, Development of Electroless Ni–P–$Al_2O_3$ and Ni–P–$TiO_2$ Composite Coatings from Alkaline Hypophosphite Gluconate Baths and their Properties, *Int. J. Electrochem. Sci., 8* 2013 1722 - 1734.
[11] A. Grosjean, M. Rezrazi, and P. Bercot, Some morphological characteristics of the incorporation of silicon carbide SiC particles into electroless nickel deposits, *Surface and Coatings Technology 130* 2000 252-256.
[12] M. Momenzadeh and .S Sanjabi, The effect of $TiO_2$ nanoparticle codeposition on microstructure and corrosion resistance of electroless Ni–P coating, *Materials and Corrosion No. 7* 2012, 63.
[13] N. K. Shrestha, D. B. Hamal, and T. Saji, Composite plating of Ni–P–$Al_2O_3$ in twosteps and its anti-wear performance, *Surface and Coatings Technology 183* 2004 247–253.