Study on influence of Surface roughness of Ni-Al$_2$O$_3$ nano composite coating and evaluation of wear characteristics

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Abstract. Electrodeposition is one of the most technologically feasible and economically superior techniques for producing metallic coating. The advancement in the application of nano particles has grabbed the attention in all fields of engineering. In this present study an attempt has been made on the Ni-Al$_2$O$_3$ nano particle composite coating on aluminium substrate by electrodeposition process. The aluminium surface requires a specific pre-treatment for better adherence of coating. In light of this a thin zinc layer is coated on the aluminium substrate by electroless process. In addition to this surface roughness is an important parameter for any coating method and material. In this work Ni-Al$_2$O$_3$ composite coating were successfully coated by varying the process parameters such as bath temperature, current density and particle loading. The experimentation was performed using central composite design based 20 trials of experiments. The effect of process parameters and surface roughness before and after coating is analyzed on wear rate and coating thickness. The results shown a better wear resistance of Ni-Al$_2$O$_3$ composite electrodeposited coating compared to Ni coating. The particle loading and interaction effect of current density with temperature has greater significant effect on wear rate. The surface roughness is significantly affected the wear behaviour and thickness of coating.

Keywords: Ni-Al$_2$O$_3$ nano-composite coating, Aluminium6061, Surface roughness, coating thickness and wear rate.

1. Introduction
Contact or mating surface of any engineering components like Cams, Drill bits, cutting tools, fasteners, piston and piston rings, automobile body parts, building materials like beams, columns, electric panels, Micro Electro Mechanical components (MEMS) etc. plays a major role in accurate functioning of the machine or system. Coating is a film of metal or non metal or ceramic produced on a surface of metal
generally stated as substrate to enhance the tribological and mechanical properties. Metal-Matrix Composites (MMC’s) can be produced by inducing Oxide or Carbide particles as second phase [1–4]. At present Nanocomposite coatings have got higher attention due to its excellent tribological, mechanical, corrosion resistant properties. In addition they exhibit higher hardness and lower coefficient of friction. These have been extensively investigating in bulk for the wide range of applications through Automobile, mills, textile, food, household products and aviation industries. But producing Nanocomposite coatings by using the traditional methods is difficult as nano particles are inert in nature, to incorporate these particles, the traditional methods have modernized and many new techniques are invented. Composite electrocodeposition has been considered as feasible method of coating due to its advantages in controlling process parameters. Composite coating mainly depends on the surface roughness and its preparation. Neseli et al.[5] Studied on influence of surface roughness of tool geometry in turning of steel base material. Sharma et al.[6] Carried out the work on finding the effect of surface roughness value Rₐ after he coating and concluded that higher values of Rₐ for the coatings.

In the present work an attempt is made to study the influence of process parameters on wear rate of Ni-Al₂O₃ nano composite coating. However, a review of the existing literature revealed that the effect of surface roughness value on the coating thickness and wear rate is unaddressed. Thus the present paper is formulated into optimization of coating parameters on the wear rate and corresponding effect of surface roughness before and after coating. Moreover the coating is characterized with the help of SEM and EDX for the composition and microstructure analysis.

2. Experimental details
2.1 Substrate preparation
The substrate material is prepared as per ASTM G99 standard which is 10mm diameter and 30mm height for wear. The substrate is polished mechanically using different grit sizes of Sic in emery paper. Then the substrate is cleaned in acetone. Then the substrate is degreased for 1-5 min, eroded in NaOH solution. In light of improving the adhesive strength of coating, the aluminium substrate is subjected to zincating process twice before electrodeposition process [7].

2.2 Electrodeposition of Ni-Al₂O₃ nano composite coating
Nickel electrodeposition was performed as per standard Watts bath solution with the following composition: nickel sulphate (NiSO₄ .6H₂O) 240 g.L⁻¹; nickel chloride (NiCl₂ .6H₂O) 45 g.L⁻¹ and boric acid (H₃BO₃) 30g.L⁻¹ [7–15]. In the present work Ni of purity 99.98% is taken as anode and substrate Al is taken as cathode. In the present work, temperature, current density, and percentage of particle concentration are considered as influencing parameters in five levels as shown in Table 1. The optimization of coating parameters is designed by central composite method (CCD). The α value considered is -1.682 and +1.682 as two axial points and at 95% of significance level. The range of experimental values and encoded levels are shown in Table 1. During the electrodeposition process the stirring speed and pH value for electrodeposition is maintained at 200rpm and 4 respectively.
Table 1. Experimental range of the variables studied using CCD

| Parameters                     | Symbols coded | Encoded values of coded levels |
|--------------------------------|---------------|--------------------------------|
|                                |               | -1.682 | -1 | 0 | 1 | 1.682 |
| Temperature (°C)               | X₁            | 30     | 34 | 40 | 46 | 50    |
| Current density (A/dm²)        | X₂            | 1      | 1.2 | 1.5 | 1.8 | 2    |
| Particle concentration (g/L)   | X₃            | 0      | 1.2 | 3  | 4.8 | 6    |

Wear test were conducted for composite coating using Pin-on-disc apparatus. The wear parameters considered are 20N load, 1.2m/s speed, 1000m sliding distance. Further, work is carried on to find the effect surface roughness on coating thickness and wear rate of composite coating. The roughness values can be measured by using Talysurf instrument on substrate before and after coating. The values are measured as shown in the equations (1), (2) and (3). Coating thickness is measured by using coating thickness meter and it is measured on dry film thickness. Coating thickness is mainly influenced by time duration of coating.

\[
\frac{X₁+X₂+X₃}{3} = X^* \quad \text{Three values are taken on X direction,} \tag{1}
\]

\[
\frac{Y₁+Y₂+Y₃}{3} = Y^* \quad \text{Three values are taken on Y direction} \tag{2}
\]

\[
R_a = \frac{X^*+Y^*}{2} \mu m \tag{3}
\]

3. Results and Discussion

3.1 Morphology study

The fig 1 shows morphology of composite coating at different coating conditions. Fig. 1(a) shows Ni coating at 0 g/L of Al₂O₃ particles. Figures 1 (b) and (c) shows the presence and uniform distribution of Al₂O₃ particles in the composite for 3 and 4.8 g/L of particle concentration respectively. The higher particle concentration at 6g/L has lead to agglomeration of particles leading to clusters which fails the coating characteristics.
Figure 1. Scanning electron micrograph of the surface of a Ni-Al₂O₃ composite coating of nano Al₂O₃ (a) 0g/L (b) 3g/L (c) 4.8g/L and (d) 6g/L. The presence of Al₂O₃ particles in the composite coating is confirmed by EDX shown in Figure 2 (b). Further study is carried on to find out the weight percentage of element presence, for this coating at the conditions of temperature, current density and particle concentration of 34°C, 1A/dm² and 4.8g/L respectively are considered. The weight percentage of composite coating is found to be 55.31, 30.76, and 13.93 of Ni, Al, and O respectively.

Figure 2. (a) SEM image of coating at 34°C, 1A/dm² and 4.8g/L (b) EDX image showing the presence of Ni and Al₂O₃ particles.
3.2 Effect of process parameters on wear rate.
The normal probability plot is given by: error = (predicted value-actual value), shows that the residuals lie reasonably close to straight line which implies that the model is significant and errors are distributed normally (Figure 3(a)).

Figure 3. (a) Normal probability plot of the residuals (b) Main effect plot (c) Interaction effect of coating parameters on wear rate.

From the main effect plot shown in Figure 3(b), particle concentration is found to be most significant factor on wear rate followed by current density and temperature. The wear rate is minimum at lower temperature and current density. The wear rate increases with the increase in temperature due to increase in KE of particle movement. Similar conclusions are drawn in current density. The wear rate remains constant at after 1 A/dm², this is due to higher anodic corrosion leading to loose adsorption of particle on the coating surface. The wear rate has decreased drastically as particle concentration is increased. The minimum wear rate is achieved at 4.8g/L of Al₂O₃. The presence of nano Al₂O₃ particles in Ni matrix composite increases the hardness of the coating thus increases the wear resistance. The wear rate is more at 6 g/L of particle concentration due to agglomeration of particles in the form of clusters at different sites. From the Figure 3(c), it is evident that the interaction effect of temperature with current density is more followed by temperature- particle loading and particle loading-current density.
The contour plot for temperature - particle concentration and current density - particle concentration is shown in Figure 4 (a) and (b) respectively. It is evident that the better wear rate is achieved at lower temperature having higher particle concentration. In the interaction effect of current density - particle concentration lower wear rate is observed at higher current density and particle concentration.

3.3 Analysis of Variance
Analysis of variance (ANOVA) is a statistical technique used to check the adequacy of the developed empirical relationships. This technique is used to identify the significance of the factors and their interactions. In this, confidence was considered to be 95%. The effect of three parameters and their interaction effects were found by analysis of variance table for the wear rate shown in the Table 2. ANOVA is carried on in order to find the optimization values of coating parameters on wear rate. The coefficient of determination \( R^2 \) of the model is 92.76 %, this clearly indicates that 92.76 % of the data is in compatibility with the predicted by model. The \( R^2 \) value always varies between 0 and 1. e predicted determination coefficient is 80.58 %, it is in the reasonable agreement with adjusted determination coefficient value 89.42%. From the ANOVA table it indicates that the contribution of particle loading on wear rate is more followed by interaction effect of temperature and current density. From the ANOVA table it is observed that the linear particle concentration contributing 87.16 followed by temperature and current density.

### Table 2. Analysis of Variance of wear rate

| Source | DOF | Seq. SS | Adj SS | Adj MS | F  | P   | % Contribution |
|--------|-----|---------|--------|--------|----|-----|----------------|
| T      | 1   | 0.000009| 0.000009| 0.000009| 1.43| 0.254| 1.40           |
| C      | 1   | 0.000001| 0.000001| 0.000001| 0.18| 0.679| 0.15           |
| P      | 1   | 0.000557| 0.000557| 0.000557| 85.02| 0.30 | 87.16          |
| T*C    | 1   | 0.000046| 0.000046| 0.000046| 6.96| 0.020| 7.19           |
| T*P    | 1   | 0.000016| 0.000016| 0.000016| 2.48| 0.139| 2.50           |
| C*P    | 1   | 0.000003| 0.000003| 0.000003| 0.43| 0.524| 0.46           |
| Residual error | 7 | 0.000085| 0.000085| 0.000007|    |      | 1.09           |
| Total  | 13  | 0.000718|        |        |    |      | 100            |

**Figure 4.** Contour plot of wear rate (a) temperature-particle concentration (b) current density- particle concentration.
3.4 Effect of Surface roughness on coating thickness, wear rate.
The surface roughness plays a major role in adhesive bonding between the coating with the substrate material and in wear rate. The effects of surface roughness value on wear rate before and after coating are shown in Figure 5 (a) and (b). The wear rate is varying with respect the variations in the value of Ra. The lower wear rate is observed at 0.456µm of Ra (Figure 5(a)), due to higher thickness of coating which is evident from Figure 5 (c). The higher Ra value has not resulted in better wear rate to weaker bonding between the coating with substrate and early exposure of substrate material in wear. The value of Ra on the coating also showed significant influence on wear rate of the coating. The Coating surface roughness is normally high and due to sliding, the rough surface asperities smoothen which in turn increases the contact area that is directly linked to an increase in CoF. The better wear rate is observed for the coating having Ra value of 1.11µm. The higher value of Ra will leads to ploughing of asperities on the coating surface.

![Figure 5. Effect of surface roughness (a) Before coating on wear rate (b) After coating on wear rate (c) Before coating on thickness.](image_url)
4. Conclusion
In summary, Ni-Al₂O₃ nanocomposite coating with better wear resistance was prepared by electrodeposition process. The presence of Al₂O₃ nano particles has increased wear resistance. The effect of coating parameters at different levels certainly affects the coating and wear rate. The following observations were from the optimization of coating parameters: i) The particle concentration has major influence followed by the interaction effect of temperature and current density ii) Wear rate is good at the temperature, current density and article concentration of 34°C, 1A/dm² and 4.8g/L respectively. iii) Higher the values of Ra will leads to higher wear rate iv) The better wear rate is achieved at Ra value of 0.456μm before coating, 1.11μm after coating and higher coating thickness at 0.456μm.

References:
[1] T.S. Pradeep Devaneyan S, Electro Co-deposition and Characterization of SiC in Nickel Metal Matrix Composite Coatings on Aluminium 7075, Procedia Eng. 97, 97 (2014) 1496–1505.
[2] L. Du, B. Xu, S. Dong, H. Yang, W. Tu, Study of tribological characteristics and wear mechanism of nano-particle strengthened nickel-based composite coatings under abrasive contaminant lubrication, wear 257 (2004) 1058–1063.
[3] D.F. Susan, K. Barmak, a. R. Marder, Electrodeposited NiAl particle composite coatings, Thin Solid Films. 307 (1997) 133–140.
[4] M. Surender, R. Balasubramaniam, B. Basu, Electrochemical behavior of electrodeposited Ni – WC composite coatings, Surface & Coatings Technology 187 (2004) 93–97.
[5] S. Neşeli, S. Yaldiz, E. Türkeş, Optimization of tool geometry parameters for turning operations based on the response surface methodology, Measurement 44 (2011) 580–587.
[6] N. Sharma, N. Kumar, S. Dash, C.R. Das, R.V.S. Rao, A.K. Tyagi, Scratch resistance and tribological properties of DLC coatings under dry and lubrication conditions, Tribology Int. 56 (2012) 129–140.
[7] Y.F. Yang, Z.Q. Gong, L.Y. Deng, B.P. Luo, Y.T. Ma, Z.H. Yang, Electrodeposition of Ni-Cr alloy on aluminum substrate, J. Cent. South Univ. Technol. (English Ed. 13 (2006) 219–224.
[8] S. a. Lajevardi, T. Shahraj, Effects of pulse electrodeposition parameters on the properties of Ni-TiO₂ nanocomposite coatings, Appl. Surf. Sci. 256 (2010) 6775–6781.
[9] B. Lv, Z. Hu, X. Wang, B. Xu, Electrodeposition of nanocrystalline nickel assisted by flexible friction from an additive-free Watts bath, Surface & Coatings Technology 270 (2015) 123–131.
[10] P. Gyftou, E. a. Pavlatou, N. Spyrellis, Effect of pulse electrodeposition parameters on the properties of Ni/nano-SiC composites, Appl. Surf. Sci. 254 (2008) 5910–5916.
[11] I. Garcia, a. Conde, G. Langelaan, J. Fransaer, J.P. Celis, Improved corrosion resistance through microstructural modifications induced by codepositing SiC-particles with electrolytic nickel, Corrosion Science 45 (2003) 1173–1189.
[12] R.P. Socha, P. Nowak, K. Laajalehto, J. Väyrynen, Particle-electrode surface interaction during nickel electrodeposition from suspensions containing SiC and SiO₂ particles, Colloids Surfaces A Physicochem. Eng. Asp. 235 (2004) 45–55.
[13] A.M. El-sherik, J. Shirokoff, U. Erb, Stress measurements in nanocrystalline Ni electrodeposits, Journal of Alloys and Compounds 389 (2005) 140–143.
[14] S.S.E.A. Pavlatou, Pulse electrodeposition of Ni / nano-TiO₂ composites: effect of pulse frequency on deposits properties, Journal of Appl. Electrochem (2010) 40:1325–1336.
[15] M.E. Bahrololoom, R. Sani, The influence of pulse plating parameters on the hardness and wear resistance of nickel – alumina composite coatings, Surface & Coatings Technology 192 (2005) 154–163.