

Research Article

# Optimization of Electrodeposition Process Parameters on Wear Properties of Ni-Wc Coated Ferrous Alloy

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## Abstract

The aim of the research work was to optimize Ni-WC electrodeposition process parameters for high wear resistant coatings on ferrous bearing. Electrodeposition parameters such as electrolyte temperature (45, 50 and 55°C), stirring rate (200, 300 and 400 rpm), current density (3, 4 and 5 A/dm<sup>2</sup>), WC concentration in electrolytic bath (1, 2, 3g/l), wear load (15, 20, 25 N) and speed (60, 90, 120 rpm) were conducted for optimization based on Design of Experiments (DOE). ANOVA results showed the percentage contribution of influencing parameters for Ni-WC coatings as WC (45.96%), stirring rate (16.65%), current density (1.92%), temperature (1.6%), load (10.00 %) and speed (11.72%). According to response surface methodology, stirring rate 262.62 rpm, temperature 55 °C, current density 5 A/dm<sup>2</sup>, WC particles 4 g/l, load 24.19 N and speed 60 rpm are the optimized parameters for the high resistance of wear and low friction.

**Keywords:** Electrodeposition, Wear Resistant Coating, Bearings, Design of Experiments, ANOVA.

## 1. Introduction

Electrodeposition composite coatings find applications in oil industries, offshore platform, steel bridges, underground pipelines, etc. due to their high wear and corrosion resistance. Many researchers reported that incorporation of SiC<sub>p</sub> shows improved wear resistance than base metal increases the hardness of the composites noticeably and the plastic deformation extent is reduced under friction condition. The composites coating with graphite contents were drastically reduced the friction coefficients. It is also observed that composites with higher content of graphite attain a steady value in shorter duration and show smaller fluctuation (Haijum Zhao *et al*, 2007). It is reported that the friction and wear behaviors of NiCeO<sub>2</sub> composite coatings are related to CeO<sub>2</sub> content. NiCeO<sub>2</sub> with CeO<sub>2</sub> content of 2.3 wt. % shows a slight increase in wear resistance compared to pure nickel coating, and coatings with higher particle content show much better wear resistance than pure nickel coatings (Yu-Jun Xue *et al*, 2006).

In addition to it, current density is also found to influence the wear resistance of the composite coatings. The wear resistance increased due to the presence of WC particles in Cr-WC coatings upto 8 A/dm<sup>2</sup> current density. Further increase in current density causes the wear surface to be coarse, and decrease in smoothness (REzaei-Sameti M *et al*, 2012). Also, particle size plays a significant role in deciding

the wear resistance of the coating film. The decrease of particle size will result in the reduction of wear loss (Narasimmana P *et al*, 2011). It is seen that addition of tungsten to nickel enhances the wear resistance of the coating layer (Haseeb A S M A *et al*, 2008). The incorporation of diamond particles into Ni-W alloy enhances the micro-hardness of the coating. Thus, material strength is improved and wear loss and coefficient of friction is significantly reduced (Haufeu Zhou, *et al*, 2005). Grain size of Ni-W, particle size of WC and its distribution, microstructure induced are the three factors which decide the hardness of the co-deposited Ni-W-WC composite coatings (Chao Guo *et al*, 2008).

Review of literature (M.Surender *et al*, 2004; C.T.Kunioshi *et al*, 2008; N. Eliaz, *et al*, 2005 ) indicated that most of the authors have focused on developing composite coatings by electrodeposition process for higher wear using different materials. Investigations on optimization of electrodeposition parameters of Ni-WC composite coatings are scarce. The objective of the present study is to optimize the electrodeposition process parameters such as current density, electrolyte temperature, stirring rate, concentration of WC particles to develop Ni-WC composite coatings for bearing applications.

## 2. Experimental Studies

Stainless steel bearing cups, used in the assembly of bicycle axles were fitted in the axle of the bearing for carrying out the wear test on customized wear tester.

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The bearing cups were polished with #1200 emery papers after cleaning these with soap, water and ultrasonically degreasing with acetone. Further, the specimens were rinsed with 5% sulphuric acid at room temperature for 5-6 minutes and finally cleaned with double distilled water. As inner surface of the bearing cup was to be coated with Ni and Ni-WC coatings.

**Table 1** Chemicals used in the preparation of electrolyte

Nickel Sulphate	Nickel Chloride	Boric Acid	Tungsten Carbide	Sodium Hydroxide
250 g/l	35 g/l	40 g/l	2, 3 & 4 g/l	6-10 ml/l

Preparation of electrolyte is done as per ASTM B689-97 standards and chemicals are used given in Table 1 and 500 ml of double distilled water were used to prepare the aqueous solution or electrolyte for each experiment. Reinforcement phase WC of 2-3 μm particle size was used in the bath. In the beginning, 50% of the water was taken into a 500 ml capacity beaker and then WC powder was added to it at concentrations of 2, 3 and 4 g/l depending on different experiments. Ultrasonicator was used to agitate the mixture for 60 min to make the WC particles sufficiently wet and to remain suspended uniformly in the distilled water. Meanwhile, remaining water is transferred to the beaker placed on the hot plate stirrer to get heated and nickel sulphate hexahydrated, nickel chloride hexahydrated and boric acid was added in a quantity mentioned in Table 1 to prepare electrolyte. After sonication, the mixture was transferred to the electrolytic bath. The pH value was set at 3.7 by sodium hydroxide (NaOH) solution. Digital pH measuring instrument was used to measure pH.

Specimens covered with mask tape act as cathode on to which coating was to be done. Pure nickel metal plate was used as anode. Both anode and cathode were inserted into the electrolyte. Distance between the two electrodes was adjusted to 90 mm for each experiment. The electrolyte was stirred continuously for different values (250, 300 & 350 rpm). Electrolyte temperature (45, 50 & 50°C) and current density (3, 4 & 5 A/dm<sup>2</sup>) were also set to the desired value. As temperature reaches the desired value, electrodeposition equipment was switched on and current was set to the required value. The experiment was carried out for 60 min agitating the solution by means of stirrer throughout the experiment to avoid settling down of WC particles. After coating, the specimens were taken out of the bath and were washed with water to remove the loosely adsorbed WC particles from the coating surface. The specimens were weighed after removing the mask tape and weight of each specimen was noted down. This was the weight of specimens before wear test.

The instrument used for measuring thickness of the coatings is manufactured by Elektro Physik was used to measure the thickness of the coated layer of Ni and Ni-WC for all specimens. The pointer of the gauge

having magnetic sensitivity was pressed against the specimen to measure layer thickness. Digital indicator displayed the value of the thickness in micrometers. The same procedure is repeated for all specimens.

The friction tests were carried out on the customized friction test conditions. Composite coated specimen i.e., ball race or bearing cup of standard size (BIS) and steel balls of 6 mm diameter were used to carry out the tests. The bearing cups and steel balls were cleaned ultrasonically using acetone and weighed by means of electronic weighing balance. Each test was performed according to DOE for 3 different loads and rotation speeds. Test duration was set to 10 min. Specimens were again cleaned ultrasonically using ethanol after completion of the each test. The test procedure is repeated for all the specimens. New steel balls were used to carry out each test. Bearing cups were weighed again to an accuracy of 0.1 mg to detect wear loss using digital weighing balance. The measurement of weight loss was done using digital weighing balance manufactured by AFCOSET balances. The least count of the equipment is 0.1mg.

### 3. Results and Discussion

The experiments are designed by statistical software Minitab V16. Design of experiments is a series of tests, in which changes in input variables are made purposefully at the same time to observe the effects on responses. The L<sub>27</sub> orthogonal array, observed response i.e., wear loss are mentioned in Table 2. According to DOE, 27 experiments are required with six parameters each having 3 levels. Main factors interactions were neglected. Stirring rate (A), concentration of WC particles (B), temperature (C), current density (D), Load (E) and speed (F) are the factors with levels 1,2,3 being (200,300,400 rpm), (2,3,4g/l), (45, 50 ,55°C), (3, 4, 5 A/dm<sup>2</sup>), ( 15,20,25 N) and (60,90,120 rpm) respectively. The responses obtained are mentioned below in Table 2. The minimum wear loss obtained was .0027 g for experiment number 25 for the levels of stirring rate (300 rpm), concentration of WC (4 g/l), temperature (55°C), current density (5 A/dm<sup>2</sup>), load 15N and speed (60 rpm).

**Table 2** L<sub>27</sub>, wear mass loss for Ni-WC composite coatings

Exp. No	Main Factors						Wear Loss gms
	A	B	C	D	E	F	
1	1	1	1	1	1	1	0.0084
2	1	1	1	1	2	2	0.0099
3	1	1	1	1	3	3	0.0141
4	1	2	2	2	1	1	0.0051
5	1	2	2	2	2	2	0.0069
6	1	2	2	2	3	3	0.0114
7	1	3	3	3	1	1	0.0043
8	1	3	3	3	2	2	0.0056
9	1	3	3	3	3	3	0.0065
10	2	1	2	3	1	2	0.0071
11	2	1	2	3	2	3	0.0077
12	2	1	2	3	3	1	0.0068

13	2	2	3	1	1	2	0.0063
14	2	2	3	1	2	3	0.0070
15	2	2	3	1	3	1	0.0047
16	2	3	1	2	1	2	0.0043
17	2	3	1	2	2	3	0.0058
18	2	3	1	2	3	1	0.0043
19	3	1	3	2	1	3	0.0079
20	3	1	3	2	2	1	0.0063
21	3	1	3	2	3	2	0.0095
22	3	2	1	3	1	3	0.0051
23	3	2	1	3	2	1	0.0062
24	3	2	1	3	3	2	0.0059
25	3	3	2	1	1	3	0.0027
26	3	3	2	1	2	1	0.0039
27	3	3	2	1	3	2	0.0051

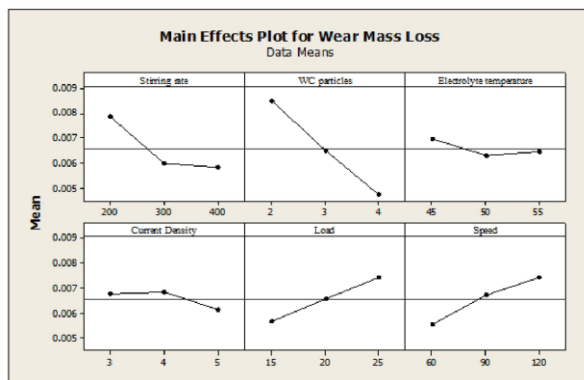


Fig. 1 Main effect plot for wear mass loss obtained from Minitab V16 for Ni-WC composites

As the temperature increased up to 50°C, the mass loss due to wear decreased. This is because the WC particles deposited in the coated layer were higher at this temperature. Also, the distribution of WC particles were uniform. Further increment in temperature will agglomerate the WC particles and may lead to more wear loss (J. Panek et al, 2011). According to author (A. Abdel Aala et al, 2009), vol. % of SiC obtained is highest at 50°C. The WC particles decreased with increase in temperature beyond 50°C, which is similar to that observed in the case of Ni-SiC. For temperatures above 60°C, desorption forces become prominent in the initial stages resulting in reduced surface concentration of nickel ions thereby reducing the coated layer. At cathode, a violent hydrogen evolution reaction has taken place at temperatures lower than 45°C and that consumed the available current density, thus making the thickness of the deposit thin.

In the present research of Ni-WC composite coatings, wear mass loss was decreased as the stirring rate increased. This is in good agreement with (Jingmao Zhao et al, 2008) wherein as the diamond particles incorporation increased up to stirring rate of 480 rpm. Again the wear loss decreased as the current density increases. Author (Leandro Trinta de Farias et al, 2008) reported that maximum incorporation of SiC particles is obtained at current density 5 A/dm<sup>2</sup>. Deposits were obtained at current densities higher than 5 A/dm<sup>2</sup> and tended to spall off at the edges of the cathode surface, mostly because of the built-in high internal stress in the coated film. Moreover, at higher current densities, a larger number of byproducts, hydrogen evolved on the cathode interface cuts down the current efficiency and deposition rate (Myong Jin Kim et al, 2008). The hardness of composite coatings is influenced by the amount of incorporated hard particles, which increases the hardness of composite coatings (M. Keddam et al, 1994). Hence, wear loss decreased as the WC particle loading in the electrolyte increased which in turn increases the WC content in the coating. Also, wear mass loss increased as the load and speed in the friction test increased. Hence minimum wear rate was observed at minimum values of load and wear test.

The main effect plot for wear mass loss that was obtained from Minitab V16 software is shown in Fig. 1. The various plots obtained for parameters against wear loss are self- explanatory.

From Table 2 it is observed that the incorporation of WC particles in Ni coatings gives minimum wear loss compared to pure Ni coatings. Hence, Ni-WC composite coatings are investigated further for the optimization of parameters.

The important factors that affect the response output were determined using ANOVA to improve the quality of composite coated layer and reduce wear mass loss. The process parameters of electrodeposition method such as temperature, current density, WC content and stirring rate and load and speed of friction test that influence response variable i.e., wear mass loss were investigated by means of ANOVA. ANOVA provides clear picture of how the process parameters affect the response and the level of importance of the factors considered. MINITAB V16 software was used to perform the above analysis. The ANOVA results are presented in Table 3 for Ni-WC composite coatings. Table 3 represents percentage contribution of each factor which influences the deposition and wear mass loss of a Ni-WC composite coatings. It is the ratio of squares of each factor to the total sum of squares of all factors.

Table 3 ANOVA for wear mass loss of Ni-WC composite coatings

Source	DOF	Sum of Squares	Mean Squares	F	P	Contribut ion
Stirring Rate	2	0.0000233	0.0000117	9.64	0.002	16.65
WC particles	2	0.0000643	0.0000321	26.59	0.000	45.96
Temperature	2	0.0000023	0.0000011	0.94	0.415	1.60
Current Density	2	0.0000027	0.0000013	1.11	0.357	1.92
Load	2	0.0000140	0.0000070	5.81	0.015	10.00
Speed	2	0.0000164	0.0000082	6.78	0.009	11.72
Error	14	0.0000169	0.0000012			12.08
Total	26	0.0000139				

The value of R<sup>2</sup> for developed empirical model for wear mass loss was 87.90 %. The high R<sup>2</sup> value indicates the better adequacy of the model. F value was calculated as the ratio of mean square of factors to the error mean square. F value indicates significance of the factors is in deciding the response variable i.e., wear mass loss. In this case, WC content factor with 0.000 P value is more

significant. The contribution of each factor in percentage is mentioned in the last column of Table 3. The percentage contribution of parameters is shown in Fig. 2.

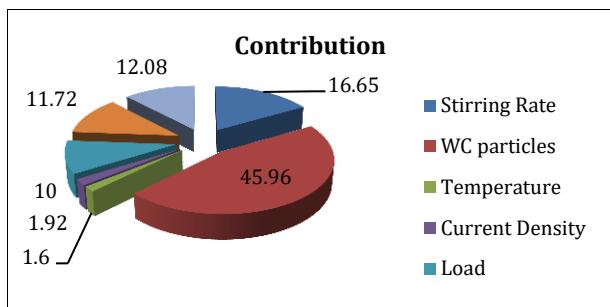


Fig. 2 Percentage contribution of parameters on Ni-WC composite coatings

**Optimization of process parameters for wear loss of Ni-WC composite coatings**

The regression equation for wear mass loss obtained from Minitab V16. The R<sup>2</sup> value obtained by means of ANOVA is 87.90%. The values of R<sup>2</sup> suggests the confidence level of regression. In this case, the R<sup>2</sup> values represent excellent correlation between experimental values. Optimizing the process parameters is important as it improves the productivity. A unique and powerful optimization procedure is developed by Desirability- based optimization technique. In the present study, desirability approach based on the RSM is used for optimizing the response variable. In this approach, the measured response is transferred to the scale of dimensionless desirability value 'd'. The function scale ranges between d=0 and d=1, where d=0 suggests completely unacceptable response and at d=1, the exact target is achieved. The optimization analysis was performed using Minitab V16. The response surface optimization parameters are shown in Fig. 3.

Optimal D	High Cur	Stirring	WC parti	Electrol	Current	Load	Speed
0.91993		400.0	4.0	55.0	5.0	25.0	120.0
		[262.6263]	[4.0]	[55.0]	[5.0]	[24.1919]	[60.0]
		200.0	2.0	45.0	3.0	15.0	60.0
Composite Desirability							
0.91993							
Wear Mas							
Minimum							
y = 0.0011							
d = 0.91993							

Fig. 3 Response surface optimization parameters of Ni-WC composite coatings

The optimized values obtained for parameters are 262 rpm stirring rate, 4g/l WC particles, 55°C temperature, 5 A/dm<sup>2</sup>, load 24 N and speed 60 rpm. The wear loss obtained by taking the above parametric values was 0.000619 g.

**Conclusions**

The electrodeposition of composite coatings was carried out to investigate wear behaviour for bearing applications. To optimize the electrodeposition parameters, an L<sub>27</sub> array for Ni-WC composite coatings, Design of experiments (DOE) and ANOVA were performed, considering stirring rate, WC particles, temperature, current density, load and speed as influencing parameters and wear loss of Ni and Ni-WC coatings as response variables. The results are discussed as follows:

- The incorporation of WC particles in Ni coatings increases the wear resistance, hence wear loss is lesser compared to pure Ni coatings which is in good agreement with previous research works. The minimum wear loss obtained for Ni coatings and Ni-WC coatings are 0.0061g and 0.0027 g respectively.
- The ANOVA result shows that wear loss for Ni-WC composite coatings is influenced by stirring rate (16.65%), WC particles (45.96%), temperature (1.6%), current density (1.92%), speed (11.72%) and load (12.08%)
- At lowest wear loss, the optimum test condition is obtained. The levels are A3B3C2D1E1F3 for Ni-WC composite coatings i.e., stirring rate (400 rpm), WC particles (4g/l), temperature (50°C), current density (3 A/dm<sup>2</sup>), load (15 N) and speed (120 rpm).
- The minimum wear loss for Ni-WC coatings provided by response surface methodology (RSM) is 0.00126 g which is almost in agreement with the experimental results

**References**

Haijum Zhao, Lie Liu, Wenbin Hu, Bin Shen (2007), Friction and wear behavior of Ni-graphite composites prepared by electroforming, Materials and Design, Vol. 28, pp.1374-1375.

Yu-Jun Xue, Xian-Zhao Jia, Yan-Wei Zhou, Wei Ma, Ji-Shun Li(2006), Tribological performance of Ni-CeO<sub>2</sub> composite coatings by electrodeposition, Surface and Coating Technology, vol. 200, pp.5677-5681.

Rezaei-Sameti M, Nadali S Rajabi J, Rakhshi M(2012), The effects of pulse electrodeposition parameters on morphology, hardness and wear behavior of nanostructure Cr-WC composite coatings, Journal of Molecular Structure, vol.1020, pp.23-27.

Narasimmana P, Malathy P, Ushpavanam, Periasamyb V M (2011), Synthesis characterization and comparison of sediment electro-codepositite nikel-micro and nano SiC composites, Applied Surface Science, vol. 258, pp.590-598.

Haseeb A S M A, Albers U, Bade K (2008), Friction and wear characteristics of electrodeposited nanocrystalline nikel-tungsten alloy films, Wear, vol. 264, pp.106-112.

Haufeu Zhou, Nan Du, Liwei Zhu, Jianku Shang, Zhouhai Qian, Xiaoming Shen(2005), Characteristics investigation of Ni-diamond composite electrodepostion, Electrochimica Acta, vol. 151, pp.157-167.

- Chao Guo, Yu Zuo, Xuhui Zhao, Jingmao Zhao and Jinping Xiong (2008), The Effects of Electrodeposition Current Density on Properties of Ni-CNTs Composite Coatings, *Surface & Coatings Technology*, 202, pp. 3246–3250.
- M.Surender, B.Basu and R.Balasubramaniam (2004), Electrochemical Behavior of Electrodeposited Ni-WC Composite Coatings, *Surface & Coatings Technology*, Vol 187, pp. 93–97.
- C.T.Kunioshi, O.V.Correa and L.V.Ramanathan (2008), High Temperature Erosion-oxidation Behavior of Thermal Sprayed Nickel-Chromium and Chromium Carbide Coatings, *International Conference on Aerospace Science and Technology*, Vol 84, pp. 1-4.
- N. Eliaz, T.M. Sridhar and E. Gileadi (2005), Synthesis and Characterization of Nickel Tungsten Alloys by Electrodeposition, *Electrochimica Acta*, Vol 50, pp. 2893–2904.
- J. Panek, B. Bierska-Piech, M. Karolus (2011) The Corrosion Resistance of Zinc-Nickel Composite Coatings, *Journal of Achievements in Materials and Manufacturing Engineering*, Vol 45/2, , pp. 157-162.
- A. Abdel Aala, H.A. Gobran and F. Muecklich (2009), Electrodeposition of Ni-RuAl Composite Coating on Steel Surface, *Journal of Alloys and Compounds*, Vol 473, pp. 250–254.
- Jingmao Zhao, Yu Zuo, Xuhui Zhao, and Jinping Xiong (2008), The Effects of Current Density on Ni-CNTs Composite Coatings, *Surface and Coatings Technology*, Vol 202, pp. 3246-3250.
- Leandro Trinta de Farias, Aderval Severino Luna(2008), Influence of Cathodic Current Density and Mechanical Stirring on the Electrodeposition of Cu-Co Alloys in Citrate Bath, *Materials Research*, Vol. 11, , pp. 1-9.
- Myong Jin Kim, Joung Soo Kim, Dong Jin Kim and Hong Pyo Kim (2008) Electro-deposition of Oxide-dispersed Nickel Composites and the Behavior of their Mechanical, *Metals and Materials International*, vol. 15, pp. 789-795.
- M. Keddad, S. Senyarich, H. Takenouti and P. Bernard (1994), A Composite Electrode for Studying Powdered Electroactive Materials Preparation and Performance, *Applied Electrochemistry*, Vol 24, pp. 1037-1043.