

Wear Behavior of Ni-TiO₂ Nano-Composite Coating on AISI 1022 CS by Pulse Electrodeposition

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ABSTRACT

The Ni-TiO₂ nano-coatings were prepared on the AISI 1022 carbon steel (CS) surface using the pulse electrodeposition method. The surface structure of CS was considerably reformed owing to the titanium particles presence.

The electroplating is widely used as a protection of the material surface to expand the life of vehicle components. The parameters of pulse frequency (PF - Hz), current density (CD - A/cm²) and duty cycle (DC - %) are combined into the RSM three factors and three level arrays to find the effect of the wear rate coating parameters. The Ni-TiO₂ nano-particles deposited surface structure and wear of the surface were examined by field emission scanning electron microscopy (FESEM) and wear test was conducted by pin on disc wear tester. The experimental result revealed a lower specimen wear rate of the parameter is the frequency: 20 Hz, current density: 0.2 A/cm² and duty cycle: 30 %. The ANOVA result revealed the pulse frequency and current density quadratic term are the most essential factor contributing 28.48, 29.95 % impact on wear resistance.

Keywords: AISI 1022 CS, Ni-TiO₂ nano composite coating, wear rate, RSM, SEM, ANOVA

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1. INTRODUCTION

Electroplating is more scientifically viable and economically superior methods for producing nano coatings. Electrodeposition methods have received much consideration improving the tribological characteristics of the coating. Praveen and Venkatesha used Sol-gel coating method on mild steel material by Zn-Ni/TiO₂ nano particle. The corrosion test was performed in 3.5 % NaCl solution, pH =4, temperature 300 K, cathodic current 2 A/dm², results revealed reduced corrosion loss and improved microhardness [1]. Zhang et al. produced a coating on a magnesium AZ91D alloy using electroless method. The Ni coating on the bottom side and another one on the top side is a Ni-TiO₂ nano coating. Ni-TiO₂ nano-coating surface hardness has greater than that of the electroless Ni surface coating. The Ni-TiO₂ coating's microhardness improved five times more than the magnesium alloy AZ91D [2].

The Ni-nano SiC coating fabricated under current density 5 A/dm², duty cycle 10, 50, 90 % and the frequency of 1, 10, 100 Hz. Generally the percentage of implanted SiC particles increased with decreasing duty cycle, this leads to improved microhardness [3]. The sol-enhanced method was used to prepare TiO₂ nano particles to obtain strengthened Ni-TiO₂ nano coating. In this paper compared the thermal stability and the tensile properties of the traditional Ni-TiO₂ nano coating with sol coating. They sol-enhanced Ni-TiO₂ coating has greater tensile strength than Ni-TiO₂ nano coating [4].

Thiemig and Bund examined the effect of nickel-titanium coating by electrodeposition. The current density increasing from 2 to 4 A / dm² when nano particle deposition enhanced

that leads to the microhardness rose from 354 to 390 HV. But the current density increasing to 8 A / dm², which results in a small reduction in microhardness to 375 HV [5]. Lajevardi and Shahrabi examined the adhesion of Ni-TiO₂ nano coating from Watts's type bath by changing parameter of current density and pH. The experimental data proved that is possible to enhance the deposition of nano-particles in a nanostructure nickel matrix by applying low current density and pH values [6].

Li et al. prepared Ni-TiO₂ coating on NdFeB magnet by pulse electrodeposition technique to examine the microhardness and performance of wear. Their operation condition is pulse frequency 500 Hz, duty cycle 30%, pH = 4.1. The results were revealed that Ni-TiO₂ coating have excellent magnetic corrosion and wear resistance [7]. Praveen and Venkatesha prepared Ni-TiO₂ nano coating on a Zinc substrate by sol-gel technique. This shows better corrosion resistance, low wear loss and enriched hardness of the nano coatings in the industry [8].

Chen et al. examined the impact of Ni-TiO₂ nano coating on a medium carbon steel substrate. The result revealed microhardness was 320 Hv in Ni coating considerably increased to 430 Hv in Ni-TiO₂ nano coating with a weight percentage of 3.26 and a decrease in wear loss of nano-coating by 50 % [9]. Bagheri et al. inspected the co-position of nano-particles increased by increasing nano-powder to a certain extent, further increase the nano-particles in electrolyte bath lead to a reduction in co-position of the coating by the presence of more particles resulting in agglomeration nano particle at the surface.

The wear loss of the Ni-TiO₂ nano-coating was smaller than the Ni-coated samples due to the present second phase reinforcement layer, which often served as a stable lubricant among contact surfaces [10]. Wang et al. reported an improvement in TiO₂ content from Ni-0 g/L TiO₂ to Ni-10 g/L TiO₂ nano composite coating reduced friction coefficient and weight loss following an improvement in TiO₂ concentration which increased the wear rate by an increase in TiO₂ lead porous structure decreased wear resistance [11].

Natarajan et al. established empirical relationships between frequency, current density and duty cycle to predict the nanocoating characteristics of MH, Ra, Ws and CoF by RSM [12]. Minimum research only carried out by Ni-TiO₂ nano coating on AISI 1022 carbon steel by pulse electrodeposition.

2. METHODOLOGY

Table 1. Experimental variables of the study in the central composite design

Input parameter value		
(P _F) (Hz)	(C _D)(A/cm ²)	(D _C) (%)
10	0.1	10
20	0.2	20
30	0.3	30

Table 2. Design matrix and experimental results of Ni-TiO₂ nano coating

S. No.	P _F (Hz)	C _D (A/cm ²)	D _C (%)	Specific Wear rate (mm ³ /Nm)
1	10	0.1	30	0.0008145
2	10	0.3	30	0.0005854
3	10	0.1	10	0.0005630
4	10	0.3	10	0.0008145
5	10	0.2	20	0.0008908
6	30	0.3	10	0.0007126
7	30	0.1	10	0.0005320
8	30	0.3	30	0.0007636
9	30	0.1	30	0.0007636
10	30	0.2	20	0.0008399
11	20	0.2	10	0.0008145
12	20	0.2	30	0.0005090
13	20	0.1	20	0.0009926
14	20	0.3	20	0.0007381
15	20	0.2	20	0.0007890
16	20	0.2	20	0.0006872
17	20	0.2	20	0.0006363
18	20	0.2	20	0.0007890
19	20	0.2	20	0.0006872

Table 3. ANOVA table for the specific wear rate

Variance	DF	SS	MS	F-value	% contribution
Model	9	1.431E-07	1.590E-08	270.15	
P _F	1	8.254E-09	8.254E-09	140.28	10.19
C _D	1	2.642E-10	2.642E-10	4.49	0.26
D _C	1	3.982E-10	3.982E-10	6.77	5.29
P _F * D _C	1	1.322E-08	1.322E-08	224.66	0.17
P _F * C _D	1	5.253E-09	5.253E-09	89.28	8.47
D _C * C _D	1	4.443E-08	4.443E-08	755.11	3.37
P _F ²	1	1.767E-08	1.767E-08	300.26	28.48
D _C ²	1	4.672E-08	4.672E-08	794.03	11.33
C _D ²	1	3.426E-09	3.426E-09	58.22	29.95
Error	5	1.062E-10	2.124E-11		2.49
Total	19	1.437E-07			100

R² = 0.9959, Adj. R² = 0.9922, Pred. R² = 0.9377

20 20 0.2 20 0.0007890

The Ni-TiO₂ nano coating was electrodeposited by nickel watts bath using pulse electrodeposition. In this experiment Nickel plate as anode and workpiece was used as the cathode. The workpiece were in cylindrical using diameter of 1 cm and a length of 3 cm. Ni-TiO₂ were electrodeposited from a typical Watts-type electrolyte, containing nickel sulphate (250 g/l), boric-acid (35 g/l), nickel chloride (40 g/l) and titanium dioxide (0-10 g/l). Before electroplating, the workpiece was polished to an emery paper with a fine surface finish. Table 1 displays the input parameters for the pulse plating. The coated workpiece microstructure was studied using SEM. In the analysis the workpiece surface, in the workpiece diameter can be kept for analysis the SEM samples is 1×1 cm in size. It can be carefully loaded into machine than adjusting the magnification of the SEM to calculate the coated surface grain structure. The wear test was executed with pin-on-disc wear tester, load of 10 N and speed a 1000 rpm. The experimental reading of Ni-TiO₂ nano-coating material are seen in Table 2.

3. RESULTS AND DISCUSSIONS

3.1 Specific wear (Ws)

Equation 1 is the Archard wear equation used to determine the basic wear rate of the coated substance in mm³ / N-m [12].

$$\text{Specific wear (Ws)} = \frac{\text{Wear volume}}{\text{(Applied load x sliding distance)}} \quad (1)$$

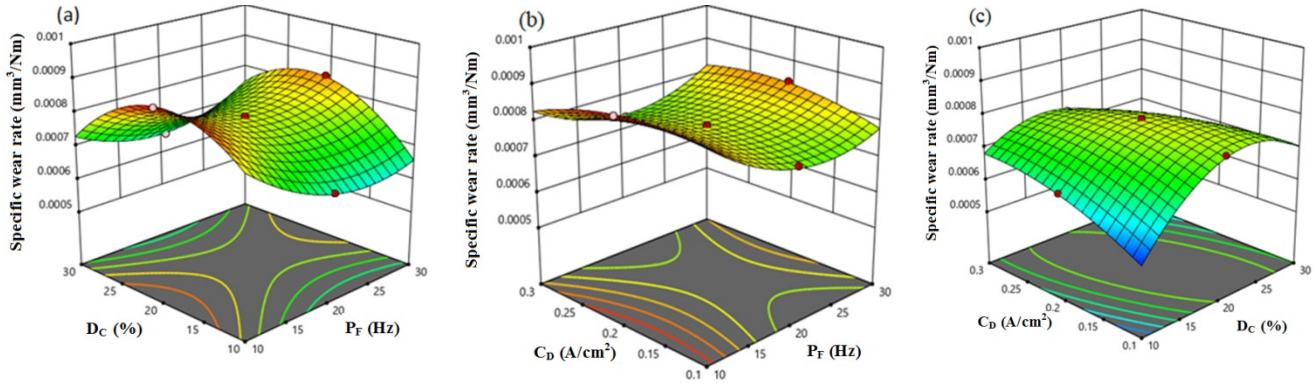
The numerical equation between pulse electrodeposition of Ni-TiO₂ nanocomposite coating parameter with specific wear rate represented in Equation 2.

$$W_s = 0.000465 - 0.000048 P_F + 0.000060 D_C + 0.002338 C_D + 4.06500E-07 P_F * D_C + 0.000026 P_F * C_D - 0.000075 D_C * C_D + 8.01545E-07 P_F^2 - 1.30345E-06 D_C^2 - 0.003530 C_D^2 \quad (2)$$

Table 3 reveals that the pulse frequency quadratic term and current density quadratic term are the most essential factor contributing 28.48, 29.95 % impact on wear resistance. The duty cycle quadratic term, the pulse frequency linear term, the interaction of frequency - current density and the linear term of duty cycle have another reasonable effect on the wear making a contribution to 11.33, 10.19, 8.47 and 5.29 % in that order. The interaction of the frequency - duty cycle and current density-frequency has the least significance on wear.

Table 4. Ranking of coating parameters

Levels	Pulse frequency (P _F) (Hz)	Current density (C _D) (A/cm ²)	Duty cycle (D _C) (%)
1	0.0007336	0.0007331	0.0006873
2	0.0007432	0.0007432	0.0007839
3	0.0007223	0.0007228	0.0006872
Max-Min	0.0000209	0.0000204	0.0000967
Ranking	2	3	1

**Figure 1.** 3D surface plot of (a) Pulse frequency (P_F)-Duty cycle (D_C), (b) - Current density (C_D)-Pulse frequency (P_F), (c) Duty cycle (D_C)-Current density (C_D) for specific wear rate

From the Figure 1(a-c) 3D plot reveals that the wear rate decreased gradually as the pulse frequency increased after a certain amount slowly increased with the pulse frequency increase. The wear rate rises with an increase in the duty cycle after a certain amount declines substantially with an increase in the duty cycles. At low frequency T_{on} is less than the T_{off} duration, which results in the removal of the weak bonding strength of the absorbed nano composite particle that results in better resistance to wear and hardness owing to hydrodynamic forces. At high frequency T_{off} duration is less than T_{on} time, the limited T_{off} period is not sufficient to remove all loosely absorbed TiO₂ particles [6].

After that the wear rate in the low duty cycle was smaller, growing with the duty cycle raising. The dwindling duty cycle contributes to a rise in the pulse T_{off} duration when the content of the TiO₂ particle deposition becomes greater than the Ni ions. The nano coated material hardness relies largely on the quantity of TiO₂ particles distributed in Ni matrix.

Strongly dispersed nanoparticles retain the drive of the nickel matrix of plastic deformation and thus raise the microhardness [6]. This reveals that electrodeposited nanoparticles play an essential role in coating wear behavior [20]. In this experiment, the wear rate is 0.0004630 mm³ / Nm with a low duty cycle of 10 %. The duty cycle is raised to 30% when wear rate was increased almost two times because of the existence of the less nanoparticle content.

At a low current density, nano-coated particles minimize the wear of nano-coated composite material. TiO₂ nanoparticles dispersion was higher in low current density than the Ni metal matrix. At higher current density results in nanoparticles being distributed less than the Ni metal matrix. From Table 4 indicates the duty cycle was influenced more on wear rate trailed by frequency and current density.

3.2 Wear behavior of Ni-TiO₂ nano coating specimen

The specimen surface structure and wear was analyzed for low wear rate specimen result is given in Table 5.

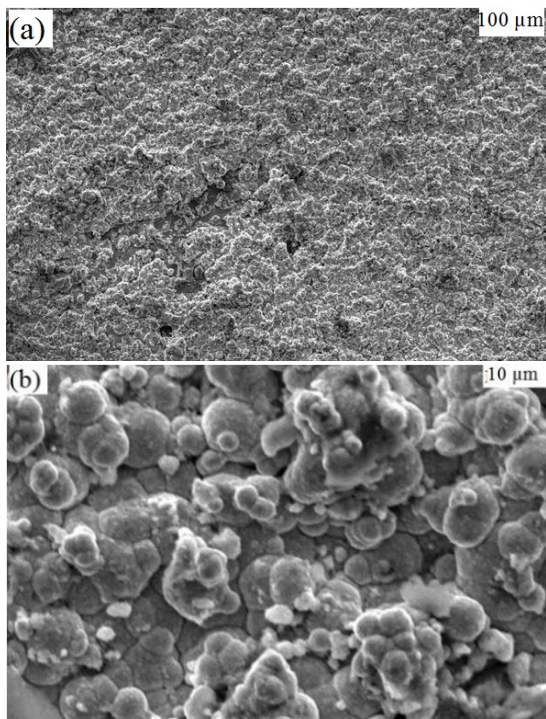
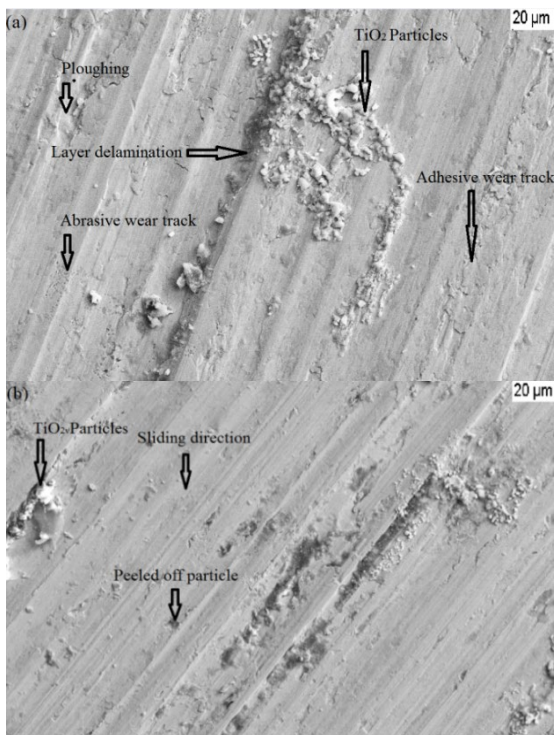
The morphologies of prepared Ni-TiO₂ nano coating characterized by SEM and the image were presented in Figure 2(a) and (b). The Ni-TiO₂ nano coating micrograph showed a better size distribution and particle growth has become more consistent at low magnification and at high magnification surface morphology is looks like dense cauliflower structure [21].

Ni-TiO₂ nano-composite AISI 1022 CS surface wear morphology shown in Figure 3(a) and (b). The heights percentage of TiO₂ accumulated on AISI 1022 steel content in the volumetric wear loss of the nano-coated specimen which has two times less than pure AISI 1022 CS sample. TiO₂ nanoparticle coated material wear surface displays adhesive wear, abrasive wear, delamination of the coating and peeling off the material. This TiO₂ nanoparticle coated substance has less wear damage owing to the resistance to touch with the surface [22].

In fact, strong nanoparticles coated in nanocomposite coatings eliminate the immediate surface interaction between the metal layer and the abrasive particles. Vaezi MR et al also stated that more nanoparticle coated content had a lower rate of wear relative to higher hardness content in nano-composite coatings. It is fairly known that subsequently the nanoparticles embedded in a nickel matrix will retain the plastic deformation of the matrix under a loading condition due to the smaller crystal size of the particles and the dispersive reinforcement of the matrix [23]. This experiment reveals lower wear rate of 0.0005090 mm³ / Nm and high wear rate of 0.0008145 mm³ / Nm and its showing more TiO₂ deposited with a finer grain size particle that prevents the plastic flow of the substance under loading and sliding conditions.

Table 5. Best experimental results of Ni-TiO₂ nano coating

Sl. No.	P _F (Hz)	C _D (A/cm ²)	D _C (%)	Specific Wear rate (mm ³ /Nm)
1.	20	0.2	30	0.0005090

**Figure 2.** (a) (b) FESEM image of Ni-TiO₂ nano coating specimen for different magnification**Figure 3.** (a) (b) FESEM image of wear surfaces of Ni-TiO₂ nano coating after wear test

4. CONCLUSIONS

The present research work concentrates the specific wear (Ws) rate with incorporating coating parameters using RSM, developed an empirical relationship which is reliable within the level of variables. This relationship can be used very effectively to predict the specific wear (Ws) rate of Ni-TiO₂ nano coating on AISI 1022 carbon steel at 95% confidence level. Based on the experimentations, the following conclusions are drawn.

(1) The ranking of coating parameters show duty cycle was more influence on nano-coating followed by pulse frequency and current density.

(2) ANOVA table shows the quadratic terms of pulse frequency and current density are the most affecting factors on specific wear rate at 28.48, 29.95 %. The duty cycle quadratic term, pulse frequency linear term, interaction of frequency - current density and the linear term of duty cycle have another reasonable effect on the wear rate.

(3) The parameter of pulse frequency: 20 Hz, current density: 0.2 A/cm² and duty cycle: 30 % Ni-TiO₂ nano coated specimen surface morphology was more uniformly distributed nano particle with cauliflower structure. The corresponding wear surface shows abrasive, adhesive, ploughing and peeled of particles.

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