SCIENTIFIC PAPER

EFFECT OF SALT BATH NITRIDING ON TRIBOLOGICAL PROPERTIES OF AISI52100 STEEL COATINGS USING RSM

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Abstract: This investigation employs the salt bath nitriding to extend the service life and improve the surface characteristics of AISI 52100, commonly employed in bearing applications. A wear test was conducted using a pin-on-disc device according to ASTM G-99 standard, and the findings show a 30% reduction in wear loss. Sliding wear experiments were conducted at 1.5 meters per sec with a 5 N force at room temperature on uncoated and nitrided pins which was obtained as optimum value from RSM. The three variables five levels central composite design (CCD) were utilized in order to reduce the number of trails and the model relations were examined through ANOVA.Surface hardness, friction coefficient, wear coefficient, Loss in wear is measured over pins without coating and nitride pins. Investigations were done into how 5W30 oil affected passive and drip lubrication. Nitride substrates had a hardness of 590 HV. Also, nitriding has a favorable effect on the friction coefficient, lowering it by up to 23%. The 5W30 lubricant will further decrease the friction coefficient. The lowest coefficient of friction was seen with the addition of 14% drip oil lubrication. Significantly less wear loss in the pin was caused by a combination of high hardness and low coefficient of friction.

Keywords: Salt bath Nitriding, Wear, Hardness, Coefficient of friction, Lubrication

Highlights

- This investigation employs the salt bath nitriding to extend the service life
- Improve the surface characteristics of AISI 52100
- Surface hardness, friction coefficient, wear coefficient, Loss in wear is measured
- Response surface methodology (RSM) design approach was performed to finding best outcomes
- Processing maps to be developed can be effectively used to identify the feasible working range.

INTRODUCTION

AISI 52100 steel is used as a bearing element predominantly in a number of applications like bearings for antifriction, cams, crankshafts, etc. [1]. The major element present in AISI 52100 steel is chromium, along with carbon, manganese, and silicon. Although the presence of chromium makes this steel more corrosive-resistant and wear-resistant, since these bearings are working relentlessly, it may undergo wear and corrode. One of the most versatile methods to protect against wear and corrosion is surface treatment [2]. A number of coating methods are available to maintain the performance of the bearing during operation [3]. The most commonly used coating process for improving the wear resistance of steel is salt bath nitriding [4]. In comparison to other surface treatment processes like carburizing and carbo nitriding, this salt nitriding process is found to be best at improving surface hardness and corrosion resistance [5].

In the salt bath nitriding process, the nitrides are deposited in the layer of steel. This nitride then reacts with chromium to form a passive layer of CrN, which is white or pale yellow. This layer is extremely hard and will not corrode or wear easily. The over hardness of the surface may lead to brittleness, so it is machined in some cases [6, 7]. The passive layer formed is in. It is thin and extremely hard. The use of lubricants is an avoidable factor in the case of bearings, so it is important to analyse the performance of surface-treated bearings with the application of lubricants [8]. One of the simplest and most reliable methods of lubrication that is commonly used is drip oil lubrication, which involves dripping oil periodically onto the bearing surfaces, creating a thin film that reduces friction and wear [9]. Passive oil lubrication is another promising method of lubrication in which the flow of lubrication is achieved without the aid of external forces [10]. Since a number of parameters are involved, optimisation of the process is important, which can be done by the RSM method due to its efficacy in producing results [11]

Nitriding is a old age technique that is followed still to improve the performance of various types of steel, a lot of work have been done in nitriding on various types of steel. Low temperature salt nitriding was done on austentic stainless steel by Chaitanya kumar et.al, The results of wear test conducted using pin-on-disc apparatus revealed that the wear rate was decreased for nitrided specimens in comparison to uncoated specimen [12]. AISI 421 martenstite steel was salt bath nitrided by Aravind et.al. Initially tempering treatments were carried out followed by salt bath nitriding. SEM image results showed uniform coating and reduced wear rate was observed on pin and disc experiment [13]. Srikanth et.al, subjected hree specimens of austentic stainless steel to salt nitriding at 570 °C for different timings of 60 minutes, 120 minutes, and 180 minutes. It was found that wear volume was reduced to a great extent after nitriding [14]. Jun Wang et al. performed salt bath nitriding on 304 austentic stainless steel. The outcomes of the experimentation revealed that the thickness of the coating improved with an increase in nitriding time. The specimens that were subjected to nitriding for 16 hours were found to have the best corrosive resistance [15].

Colombini et.al, used response surface methodology to optimize the parameters involved in nitriding process. Using RSM it was able to obtain number of surface hardness value and different wear rates for various parameters. The minimum wear rate was observed for sample which was laser quenched at 1150 °C [16]. Hamad et al. incorporated the design of an experimental statistical method to optimise the laser nitriding process. The optimised parameters were 2.84 kW of laser power, 5 mm/s scanning speed, and a 2076 l/h nitrogen flow rate. Based on the prediction, the maximum microhardness was 1920 HV0.15, but the maximum microhardness was 1382 HV0.15 [17]. In this investigation, three variables five levels central composite design (CCD) were utilized in order to reduce the number of trails and the model relations were examined through ANOVA.

Although a lot of work has been carried out in the field of salt bath nitriding for various types of steel, no literature records were found for nitriding on AISI 52100 steel along with drip and passive lubrication. This makes this research a novel contribution in the field of nitriding. The foremost aim of salt bath nitriding is to increase the AISI 52100 steel's service life and further reduce wear loss and coefficient of friction (COF) through passive and drip lubrication. This study attempts to scientifically observe the surface characteristics, such as wear and hardness, of salt-nitrided AISI steel for coated and uncoated specimens. Response surface methodology is used for optimising the number of parameters involved in order to find the best outcome.

MATERIALS AND METHODS

Sample Preparation

The material used is AISI 52100 steel purchased in the form of substrates and rods. The specimens used are shown in figure 1. The chemical composition of the steel is listed in Table 1 (a)

Table 1 to be inserted here

Using the CNC machine, from the steel rod purchased, the pins for the wear test were prepared for 8 mm diameter and 32 mm length. The specimens were initially grinded, and in order to achieve a good surface finish, polishing was done using emery paper of different granule sizes, from 240 to 2000 mesh. The ASME Y 14.5 standard was used to verify the flatness, roughness, and perpendicularity of the material.

Figure 1 to be inserted here

Nitriding

Three processes make up the nitriding process: pre-oxidation, salt bath nitriding, and cooling. Before being nitrided, the samples were pre-oxidised to 350°C in a salt bath pre-oxidation medium or an air furnace.

Nitriding process done is salt bath nitriding. It consists of three steps as pre-oxidation, salt bath nitriding and cooling. In pre-oxidation process the steel is heated in air at a temperature of 350 °C in an air furnace. A thin oxide layer is formed during pre-oxidation, which improves adhesion, enhances the diffusivity of nitrogen atoms, and reduces white layer formation. Then the substrate is dipped in a molten salt bath. The proportion of each salt in the salt bath is tabulated in Table 1 (b).

The samples are immersed in a salt bath for three hours at 565 ± 8 °C. The range of temperature for nitriding is from 500 ˚C to 700 ˚C for low temperature nitriding [18], which represents the ferritic that occur at 570 °C and 700 °C, respectively. Austenitic phase occurs above 900 °C. This is high temperature nitriding. Two types of nitridings are commonly carried out as Ferritic Nitrocarburizing (FNC) and higher temperature nitriding known as Austenitic Nitrocarburizing (ANC). In this work FNC is carried out. Finally, the specimens were cooled in water at room temperature [19].

Lubricant

5W30 oil was employed as the lubricant in this experiment. The oil has a kinematic viscosity of 63.2 m^2/s at 40 °C and 10.5 at 100 °C. It has a density of 0.859 x 10^3 kg/m^3. Before conducting wear testing, nitriding was applied over passive, and drip oil lubrication was done for about 10 minutes at room temperature. Oil lubrication reduces the heat produced by friction.

Surface characterization

Surface roughness (Ra) and Hardness

According to the ASTM E950 standard, the surface roughness was assessed using a profilometer. The roughness value was first determined after the pin's perpendicularity and flatness were verified. The micro-Vickers hardness tester was used to measure surface hardness. The hardness with respect to the depth as shown in Figure 2 (a).

Figure 2 to be inserted here

Wear

The wear performance of materials is frequently determined through testing on pin-on-disc machinery using the ASTM G99 standard approach. It provides a standard procedure for conducting sliding wear testing in laboratories. The experiments were carried out using a force of 5 N as well as a uniform sliding radius of 10 mm at a speed of 1.5 m/s which is selected as optimum combination from RSM optimization. The disc is rotated at 300 rpm, and the sliding distance is 3000 m. Load cell series were used to measure the tangential force during testing, and a computerised data-collecting system kept track of it. In each case, the average value of the pins was utilised to estimate the friction coefficient, wear loss, and wear coefficient of the pin.

Morphology and structural composition

By observing the coated specimen at various magnifications (10 μm, 20 μm, 50 μm, 100 μm, and 200 μm) in the VEGA3 TESCAN SEM equipment it was able to make the microstructural examination visible. Double-sided conductive carbon adhesive tapes were used to position and secure the specimen to the SEM holder and prevent charge buildup while it was exposed to the electron beam.

This test is also useful in determining the component's chemical composition along with the corresponding weight proportion, which is known as EDX analysis. This research will assist in improving the structural composition of the material concerning the appropriate chemical elements that are present.

The fundamental requirement in any process is the selection of appropriate input parameters for generating the best outcome. For the present study, three important parameters were considered: type of coating, sliding velocity and applied load. The output performances considered were wear and COF. The RSM approach was utilized to achieve the salt bath nitriding process on AISI52100 Steel. The levels and the machining variables and DoE are listed in **Table 2**. Optimal DOE was implemented in order to reduce the required experimental trials [20,21].

Table 2 to be inserted here

RESULTS AND DISCUSSIONS

The variation in microhardness of nitriding-coated substrates for different layers can be seen in **Figure 2**. Surface hardness values show evidence of nitriding, which is represented graphically in **Figure 3**. The values obtained are in line with those obtained by researchers in the literature for other steels that were nitride. The microstructure of the salt bath layer is depicted in **Figure 1 (b).**

Optical microscope observations revealed that 321.9 HV0.1 is the core hardness, and the yellow highlighted area (Figure 2) represents the cutoff hardness at a depth of 315 micrometers. There was a compound layer between 11 and 13 microns. The diffuse zone is also seen in Figure 2, beneath the white layer. [22]. The diffusion layer and two compound layers make up the nitriding layer [23].

The EDX analysis of the substrate shows the phases of the steel matrix as well as a group of phases that are linked to chromium and are spread out in the structures of the material. The peaks in the matrix belong to Fe, whereas the remaining phases are carbides produced by alloying elements. The EDS image and spectrum are displayed in Figure 2 (b).

According to the outcome of the energy dispersive X-ray investigation as shown in Figure 2 (b), the uncoated sample contained the key components (C, Fe, Ni, and Cr). The greatest amount of unnormalised percentage of weight concentration, normalised percentage of weight concentration, proportion of atomic weight, and weight percentage concentration inaccuracy at sigma level one are all present in the carbon element. The results also showed that the phosphorus components have the lowest atomic weight percentage, unnormalized weight concentration percentage, normalised weight concentration proportion, and concentration of weight percentage error at the sigma 1 level.

Figure 4 to be inserted here

Influence of process parameters on wear

As seen in Figure 3, the material was noticeably peeled off, along with the fissures that had developed on the uncoated specimen surface. The properties of the diffusion zone were examined in this work. A clear and distinct compound layer is visible in every SEM image, and the compound layer will have incredibly small micro-etches pits.

Figure 3 to be inserted here

Figures 3 (b) and (c) depict the thick free compound layers found in the etch pits 3 (b). When small amounts of nitrogen dissolve just below the compound region, the substrate's wear resistance is affected by the forms of nitrides that are rarely made. On the other hand, when there are higher nitrogen diffusion zones, more nitrides are made, which strengthens the wear resistance.

The nitride pin's wear track is shown in **Figure. 3 (d).** The interior of the wear track is visible with wear debris and delamination. For steady-state wear, Archard proposed the equation (1) for volumetric material loss as,

$$V = \frac{KsPL}{3H} \tag{1}$$

Where, V is the volumetric material loss

L is slide over a length L

P is perpendicular force towards the worn layer

H is the pin's Brinell hardness value

Ks is the standard wear coefficient, Ks.

Considering specific parameters of V, P, L, and H, the normal coefficient of wear can be determined using the equation (2),

$$Ks = \frac{3HV}{PL} \tag{2}$$

Volumetric wear loss can be calculated using the weight loss W and density.

The larger preliminary running-in rate of wear, according to Yang [24], will initially have a greater value inside the transitional wear phase and may gradually acquire a constant level whenever the wear loss approaches a uniform level. The wear coefficient varies as there is a change in the distance of sliding, as seen in **Figure 4 (a).** It has been found that greater sliding distance causes a decrease in the wear coefficient. Nitriding with drip oil lubrication pins, however, exhibits the lowest wear coefficient under identical circumstances. The lowest volumetric loss ever observed is the main factor. According to literature [25], the dehydrated, as well as the changed fresh surface coating, is more reactive to lubrication than the original Huralite.

Figure 4 to be inserted here

Figure 4 (a) depicts the sliding distance and wear. It reveals the wear COF of uncoated, coated, nitriding with passive oil lubrication, and nitriding with drip oil lubrication. The starting and ultimate weights of the pin are used to compute the wear loss. When nitriding, drip oil-lubricated pins show lower wear loss than uncoated pins. Table 3 shows the ANOVA for wear. From that, it is observed that the sliding velocity is the most predominant parameter that affects the surface quality. The R2 value for wear is 0.9920. The model is significant.

Figure 4 to be inserted here

Table 3 to be inserted here

From **Figure 4 (b),** it is evident that the uncoated sample will have a more significant wear loss compared to the coated specimen. Nitriding with drip oil lubrication, Nitriding with passive oil lubrication, and Nitriding coating methods were used in this experimentation. Among all the three coating methodologies, the Nitriding with drip oil lubrication methodology was observed to produce less wear loss. When compared to the surface of the AISI steel substrate, there was a significant drop. The wear markings on AISI steel are much wider than the wear marks on the other samples, which are at different widths. This is so that the AISI 52100 steel's wear resistance may be greatly increased by the nitriding surface. The correlation graphs between the predicted and actual values for wear and COF are displayed in **Figure 5 (a).**

Figure 5 (a-c) presents the 3D surface images for the wear of nitriding, nitriding with drip oil lubrication, and nitriding with passive oil lubrication. It is clearly observed that the applied load is directly proportional to the wear. Figure 5 (a) illustrates the increased wear that the nitriding pin produces. During the nitriding process, the amount of nitrogen diffused into the sample surface determines the wear resistance of the specimen. From the detailed experimental procedure, it can be inferred that when the nitriding process is aided by proper lubrication, a higher concentration of nitrogen will diffuse into the steel periphery. In relevance to the characteristics, the Nitriding process without lubrication will eventually diffuse less nitrogen than the Nitriding aided by drip and passive lubrication systems.

It can in turn result in insignificant wear resistance improvement compared to the nitriding methods with lubrication. The sliding velocity and applied load can influence the wear values of the nitrided steel. But the range of wear will be high in the plain nitride process when compared with the other methods. Out of all the three methods of nitriding investigated in this research, the drip lubrication nitriding procedure proved to be capable of depositing a greater quantity of nitrogen on the surface. This makes the specimen treated with drip lubrication nitriding have good wear properties when compared with the other two methods. The outcome can be clearly understood from Figure 5 (b)

Figure 5 to be inserted here

From **Figure 5** (c), the amount of nitrogen deposition determines the wear properties of any metal. It is understood from the inference that nitriding methodologies with lubrication can ultimately deposit a significant quantity of nitrogen on the steel surface. The amount of nitrogen sediment on the sample surface in the case of nitriding with passive lubrication will be higher than in the plain nitrogen-adding process. It can make the specimen that undergoes passive lubrication nitriding have better wear characteristics than those of the normal nitriding procedure.

Influence of process parameters on Coefficient of Friction

The necessary test protocols were used to conduct pin-on-disc testing at around 1.5 meters per second as obtained from RSM as optimized value [26]. The wear, frictional force, and time were measured under a load of 10 N at every 15 minutes of sliding. Figure 5 (g) depicts the relationship between friction coefficient and sliding distance for uncoated, nitrided, nitrided with passive oil, and nitrided with drip oil lubrication.

The difference in the COF with respect to the sliding velocity of the specimens' nitrides with different methods is detailed in **Figure 5** (g). From the investigation, it is evident that the drip lubrication nitriding method can produce significantly less COF friction than the other two methods. The specimens that are being nitrided with drip lubrication exhibit less wear loss due to their low COF.

It demonstrates the pattern with which the COF reduces with sliding distance, which is a distinctive aspect of the diagrams. The load does not affect the friction coefficient of pure metals; nevertheless, on the surface of a nitrided metal, the breakdown in the nitride layer can change the COF. By nitriding with drip oil lubrication, the friction coefficient can be reduced by 0.1. Table 4 represents the ANOVA for COF. From the table, it can be inferred that the sliding velocity is the most influential parameter that affects the quality. R^2 for COF is 0.9962, and the adequate precision is 91.1079.

Comparing the nitriding, drip lubrication nitriding, and passive lubrication methodologies, the nitriding treatment sample exhibits more COF. The nitriding process without lubrication can result in a more significant variation of COF concerning the sliding velocity and applied load. The plain nitrided samples will have a COF value of 0.5, and the value drastically changes with sliding velocity and load, as shown in **Figure 5 (d).** The drip lubrication process, including the nitriding process, can stabilise the variation of COF with respect to sliding velocity and applied load, as shown in Figure 8(e). By treating the AISI 52100 steel with a drip lubrication nitriding process, the COF can eventually be reduced, which in turn enhances the wear characteristics of the material. Comparing all three nitriding techniques, the drip lubrication nitriding procedure can restrict wear significantly with its lower COF values. During the passive lubrication nitriding process, a moderate quantity of nitrogen will be deposited over the surface of the specimen. This can restrict the wear to a maximum value of 0.3. While contrasting all three nitriding processes, nitriding with passive lubrication can eventually result in drastic variation in COF with respect to the variation of sliding velocity and applied load. The variation pattern of COF is detailed in **Figure 5 (f).**

Validation of optimizing procedures

The quadratic models are developed for all of the responses (wear and COF) to find the optimal combinations of input variables. The goal of optimization is to decrease the wear rate. Table 5 displays the outcome of the optimization parameters. The highest limits of wear and COF are 0.1029 and 0.54945, respectively. The final set of process variables is sliding velocity (1.5 m/s) and applied load (5 N).

Figure 6 to be inserted here

Table 4 to be inserted here

Figure 6 shows the various solution models created for validating the models. Five models are constructed for predicting the response, and the outcome reveals that the desirability of the produced model is greater than 0.90. A validation test was also carried out in order to validate the optimized findings, as shown in Table 4, which shows the error percentages attained after running confirmation tests. The obtained error percentages are negligible, ranging from 2.88% to 4.375%. Since the validation test is carried out using the variables specified from the previous findings and various combination sets are allocated, the results demonstrate that the features seem closely related [27–30].

CONCLUSIONS

Salt bath nitrided pins of surface roughness 0.3 were successfully tested for their friction, wear, and wear loss under a 5 N load and a sliding velocity of 1.5 meters per second. The pins were tested for four conditions: uncoated, nitrided, nitrided with passive oil lubrication, and nitrided with drip oil lubrication. The important results of these tests are as follows,

- When nitriding using drip oil-lubricated pins, the friction coefficient is significantly less when the pins are uncoated.

- In comparison to uncoated pins, nitrides pins with drip oil lubrication experienced a significant reduction in friction coefficient.
- Response surface methodology optimization was useful in optimizing the parameters involved in experimentation with low level error between actual and predicted values.
- The optimum combination solution obtained of multi-response for higher desirability is 1.5 m/s sliding velocity, 5 N applied load and the coating of nitride with drip oil lubrication which have a low wear rate of 0.01311.
- The combined effect of nitriding and drip oil lubrication leads to low friction coefficient of 0.101 is observed under 1.5 m/s and 5 N, which is attributed to the enhanced tribological properties.
- Drip oil lubrication of nitrided substrates reduced the wear coefficient to 0.00218 under 5 N load and sliding speed 1.5 m/s which signifies exceptional wear resistance.
- Nitriding with drip oil lubrication reduces the wear loss due to minimized material degradation.
- Scanning electron microscopy (SEM) provides visual evidence of the superior bonding achieved through nitriding with drip oil lubrication. This enhanced bonding plays a key role in the observed reduction in COF and wear exhibited by these pins.

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Figures captions:

Figure 1 (a) Specimens used for experimentation (b) Different Layers observed in SEM-EDS (Compound Layer, Diffusion zone and white layer)

Figure 2 (a) Micro hardness with respect to depth (b and c) EDS image and EDS pattern for Uncoated and coated AISI 52100 Pins

Figure 3 SEM image for nitride specimen

Figure 4 (a) Sliding Distance vs Wear (b) Wear Loss with respect to coating

Figure 5 (a) Correlation graphs for wear and COF (b) 3D Surface graphs of Nitriding Nitriding with drip oil lubrication Nitriding with passive oil lubrication (a-c) wear (d-f) COF (g) Coefficient of Friction Vs Sliding Distance

Figure 6 Desirability graph of variables for RSM

List of Tables

Table 1 (a) Material composition in weight percentage

C	**Cr**	**Si**	**Mn**	**S**	**P**	**Fe**
1.1	1.4	0.26	0.39	0.019	0.011	Bal

(b) Salt proportion in weight percentage

Salt	**Proportion in weight %**	**Purpose**
Potassium nitrate (KNO_3)	70	Diffusion of nitrogen
Sodium carbonate (Na_2CO_3)	20	Fluxing agent
Carbamide ($CO(NH_2)_2$	5	Adjust melting point
Borides ($Na_2B_4O_7$)	4	Enhance wear resistance
Aluminium oxide (Al_2O_3)	1	Nitride layer adhesion

Table 2 Variables, levels and RSM optimal design with experimental results

Factors/Level	Type of coating	Sliding Velocity (B) in m/s	Applied load (A) in N
-1	Coating 1 (Nitriding)	1(0.3 m/s)	5
0	Coating 2 (Nitriding+ Drip oil Lubrication)	2(1 m/s)	25
1	Coating 3 (Nitriding+ passive oil Lubrication)	3(1.5 m/s)	50

	Process Parameters			Responses	
Exp. No.	**Type of coating**	**Sliding Velocity (B)**	**Applied load (A)**	**Wear (µm)**	**Coefficient of Friction**

		in m/s	in N		
1.	1	1 (0.3 m/s)	5	0.0647563	0.54945
2.	1	1	25	0.0475563	0.446506
3.	1	1	50	0.0462875	0.282919
4.	1	2 (1 m/s)	5	0.0709812	0.3627
5.	1	2	25	0.0545875	0.370225
6.	1	2	50	0.0548187	0.341662
7.	1	3 (1.5 m/s)	5	0.02895	0.155006
8.	1	3	25	0.0124937	0.242981
9.	1	3	50	0.013475	0.31705
10.	2	1	5	0.0433562	0.315606
11.	2	1	25	0.0293625	0.357637
12.	2	1	50	0.0328	0.372287
13.	2	2	5	0.0531063	0.0856937
14.	2	2	25	0.0397187	0.239725
15.	2	2	50	0.0434875	0.3857
16.	2	3	5	0.0134562	-0.213031
17.	2	3	25	0.00074375	0.0742437
18.	2	3	50	0.0049625	0.33905
19.	3	1	5	0.00909375	0.302638
20.	3	1	25	0.0245625	0.242119
21.	3	1	50	0.0650312	0.131944
22.	3	2	5	0.0324813	0.191238
23.	3	2	25	0.0492125	0.241363
24.	3	2	50	0.1029	0.2703
25.	3	3	5	0.003725	0.0530938
26.	3	3	25	0.0214625	0.174613
27.	3	3	50	0.059175	0.325694

Table 3 ANOVA of Wear and Coefficient of Friction

ANOVA of Wear						
Source	**Σ^2**	**Dof**	**Mean Squares**	**F-statistics**	**p-value**	**Remarks**
Model	0.0152	11	0.0014	168.52	< 0.0001	significant
A-Sliding Velocity	0.0017	1	0.0017	211.55	< 0.0001	
B-Applied Load	0.0007	1	0.0007	81.29	< 0.0001	
C-Type of coating	0.0011	2	0.0006	67.07	< 0.0001	
AB	3.156E-06	1	3.156E-06	0.3844	0.5446	
AC	0.0008	2	0.0004	47.53	< 0.0001	
BC	0.0055	2	0.0028	335.54	< 0.0001	

A^2	0.0049	1	0.0049	593.73	< 0.0001	
B^2	0.0005	1	0.0005	66.46	< 0.0001	
Residual	0.0001	15	8.209E-06	**R^2**		0.9920
Cor Total	0.0153	26		**Adjusted R^2**		0.9861
				Predicted R^2		0.9728
				Adeq Precision		49.5553

ANOVA of Coefficient of Friction

Source	**Σ^2**	**Dof**	**Mean Squares**	**F-statistics**	**p-value**	**Remarks**
Model	0.5751	11	0.0523	353.05	< 0.0001	significant
A-Sliding Velocity	0.1246	1	0.1246	841.00	< 0.0001	
B-Applied Load	0.0506	1	0.0506	341.75	< 0.0001	
C-Type of coating	0.0935	2	0.0468	315.80	< 0.0001	
AB	0.1562	1	0.1562	1054.73	< 0.0001	
AC	0.0438	2	0.0219	147.71	< 0.0001	
BC	0.0940	2	0.0470	317.20	< 0.0001	
A^2	0.0107	1	0.0107	72.09	< 0.0001	
B^2	0.0019	1	0.0019	12.51	0.0030	
Residual	0.0022	15	0.0001	**R^2**		0.9962
Cor Total	0.5774	26		**Adjusted R^2**		0.9933
				Predicted R^2		0.9853
				Adeq Precision		91.1079

Table 4 (a) Conditions of output responses

Parameter	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
Sliding Velocity	Is in range	6	10	1	1	3
Applied Load	Is in range	10	30	1	1	3
Types of Coating	Is in range	Coating 1	Coating 3	1	1	3
Wear	Minimize	0.000743	0.1029	1	1	3
COF	Minimize	0.1620	0.54945	1	1	3

(b) Optimum combination solutions of multi-responses for higher desirability

No	Sliding Velocity	Applied Load	Types of Coatings	Wear	COF	Desirability

1	**1.5**	**5**	**3**	**0.01311**	**0.183**	**0.9192**	**Selected**
2	1.48	5	3	0.01315	0.187	0.9160	
3	1.5	4.8	3	0.01320	0.192	0.9140	
4	1.5	4.5	3	0.01364	0.195	0.905	
5	1.4	4.5	3	0.01370	0.198	0.895	

(c) Confirmation experiments for optimization

Variable	Settings	Responses	Prediction Value	Experimental Value	% Error
Sliding Velocity	1.5	Wear	0.01311	0.0145	2.41
Applied Load	5	Coefficient of Friction	0.1836	0.192	3.33

<u>List of Figures</u>

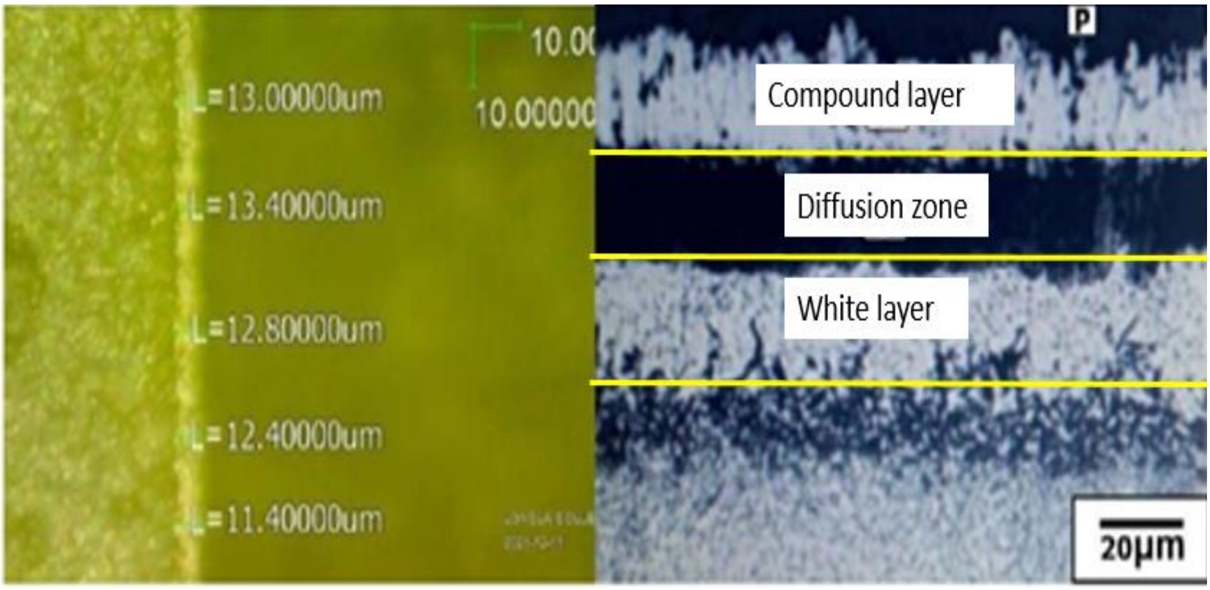
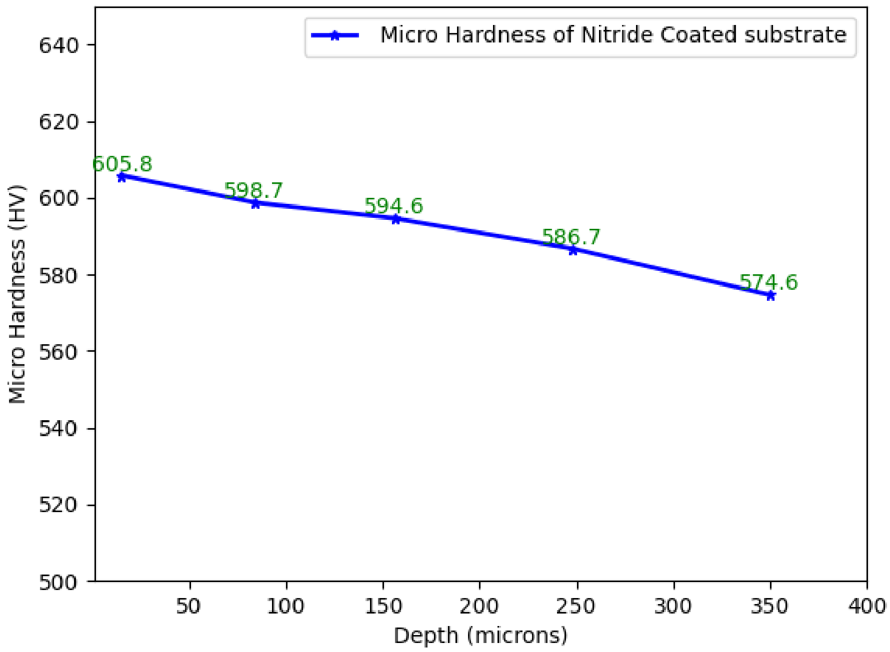

L=13.00000um
L=13.40000um
L=12.80000um
L=12.40000um
L=11.40000um
10.00000
Compound layer
Diffusion zone
White layer
P
20μm

Figure 1


Micro Hardness of Nitride Coated substrate
Micro Hardness (HV)
Depth (microns)
640
620
600
580
560
540
520
500
50
100
150
200
250
300
350
400
605.8
598.7
594.6
586.7
574.6

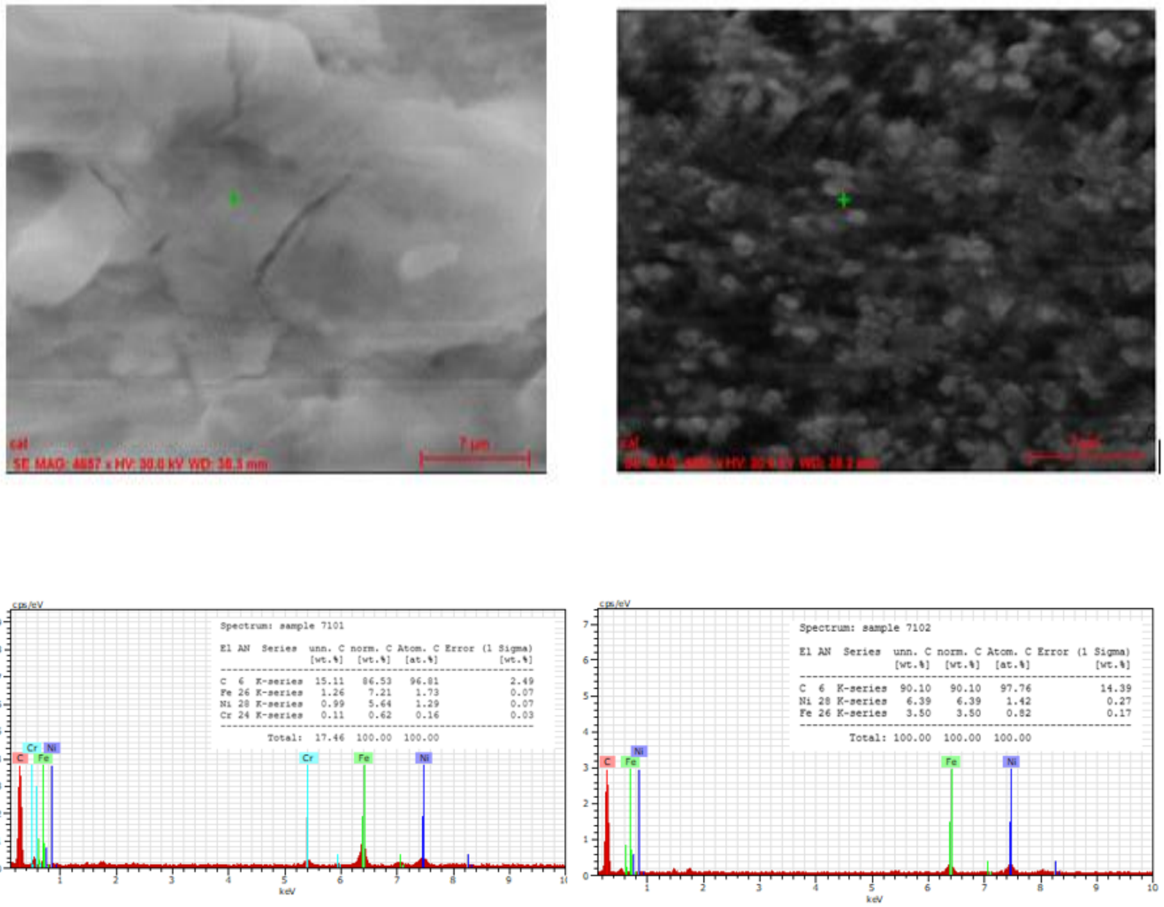

cps/eV
Spectrum: sample 7101
El AN Series unn. C [wt.%] norm. C [wt.%] Atom. C [at.%] Error (1 Sigma) [wt.%]
C 6 K-series 15.11 86.53 96.81 2.49
Fe 26 K-series 1.26 7.21 1.73 0.07
Ni 28 K-series 0.99 5.64 1.29 0.07
Cr 24 K-series 0.11 0.62 0.16 0.03
Total: 17.46 100.00 100.00
C Cr Fe Ni Cr Fe Ni
keV
cps/eV
Spectrum: sample 7102
El AN Series unn. C [wt.%] norm. C [wt.%] Atom. C [at.%] Error (1 Sigma) [wt.%]
C 6 K-series 90.10 90.10 97.76 14.39
Ni 28 K-series 6.39 6.39 1.42 0.27
Fe 26 K-series 3.50 3.50 0.82 0.17
Total: 100.00 100.00 100.00
C Fe Ni Fe Ni
keV

Figure 2

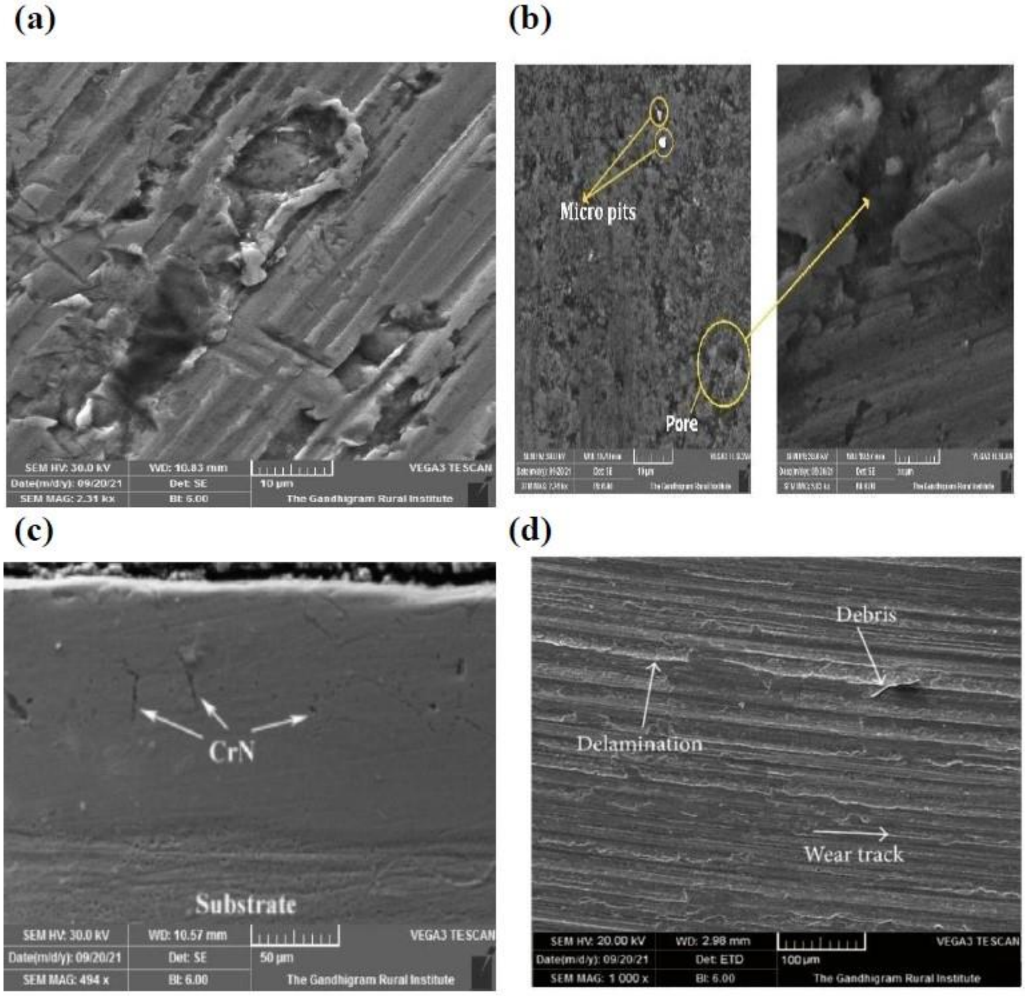
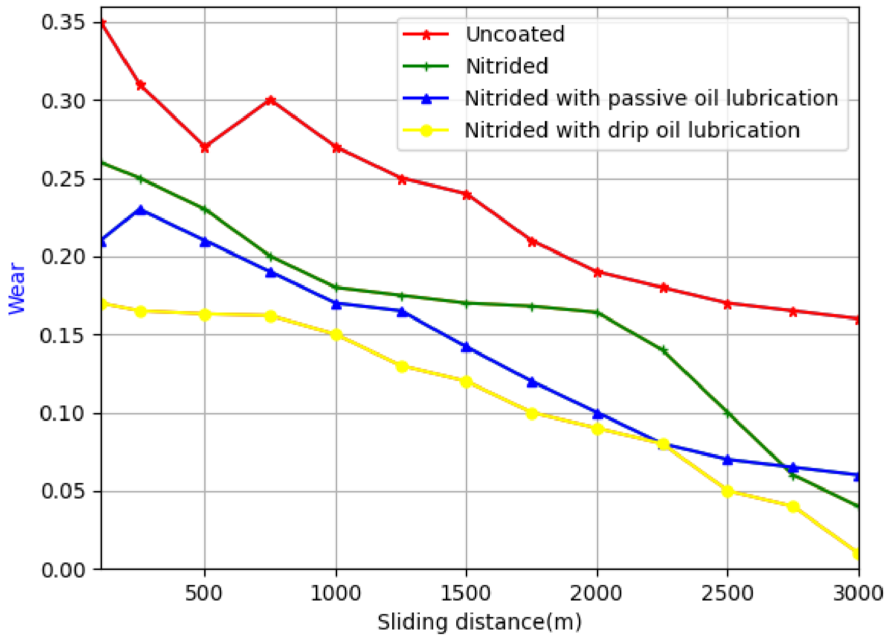
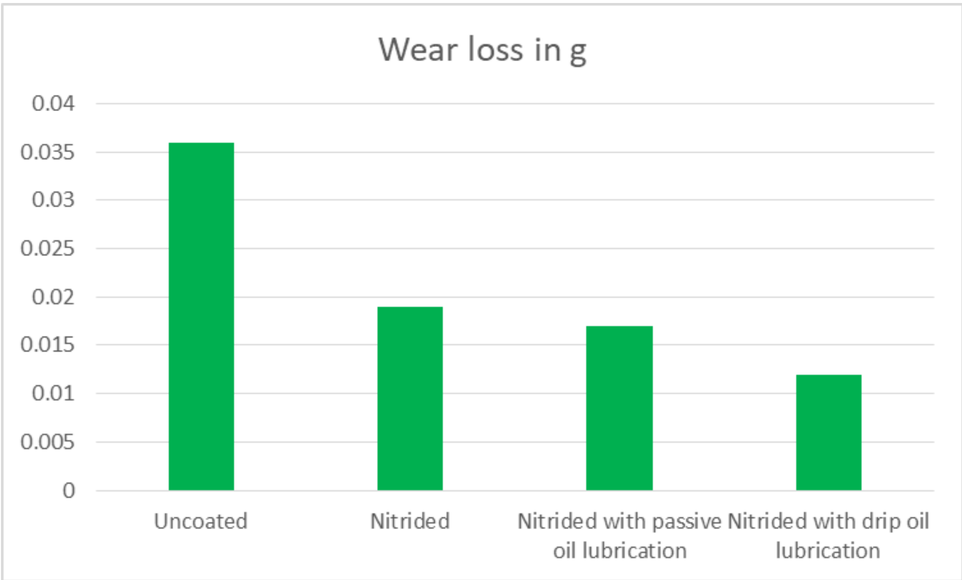

(a)
(b)
Micro pits
Pore
(c)
CrN
Substrate
(d)
Debris
Delamination
Wear track
SEM HV: 30.0 kV
WD: 10.83 mm
VEGA3 TESCAN
Date(m/d/y): 09/20/21
Det: SE
10 μm
SEM MAG: 2.31 kx
BI: 6.00
The Gandhigram Rural Institute
SEM HV: 30.0 kV
WD: 10.57 mm
VEGA3 TESCAN
Date(m/d/y): 09/20/21
Det: SE
50 μm
SEM MAG: 494 x
BI: 6.00
The Gandhigram Rural Institute
SEM HV: 20.00 kV
WD: 2.98 mm
VEGA3 TESCAN
Date(m/d/y): 09/20/21
Det: ETD
100 μm
SEM MAG: 1 000 x
BI: 6.00
The Gandhigram Rural Institute

Figure 3

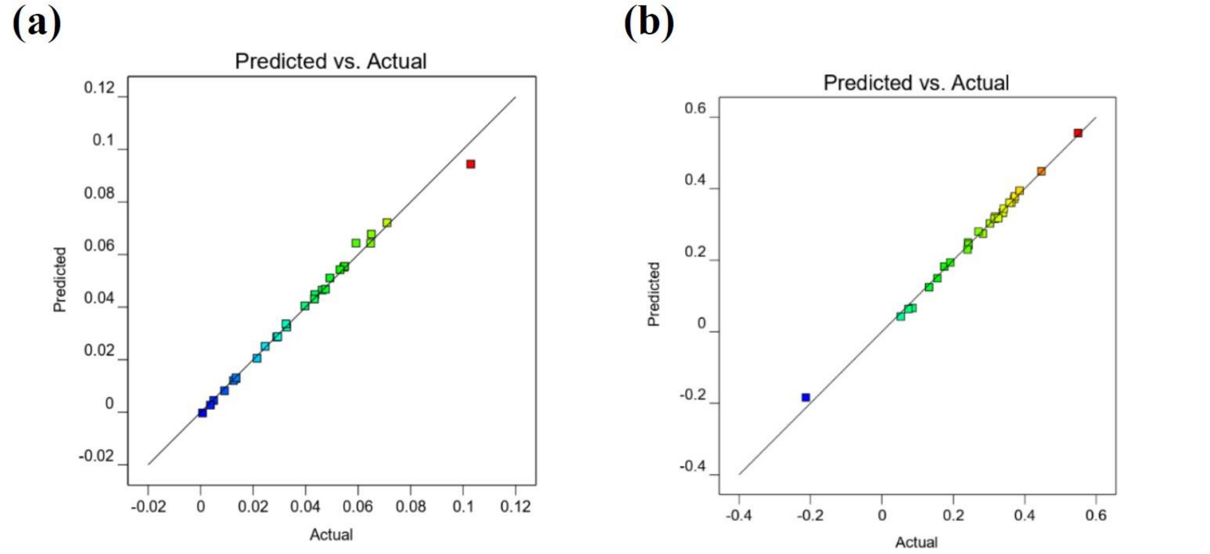

Uncoated
Nitrided
Nitrided with passive oil lubrication
Nitrided with drip oil lubrication
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
Wear
500
1000
1500
2000
2500
3000
Sliding distance(m)

(a)


Wear loss in g
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
Uncoated
Nitrided
Nitrided with passive oil lubrication
Nitrided with drip oil lubrication

(b)

Figure 4


(a)
Predicted vs. Actual
Predicted
Actual
(b)
Predicted vs. Actual
Predicted
Actual

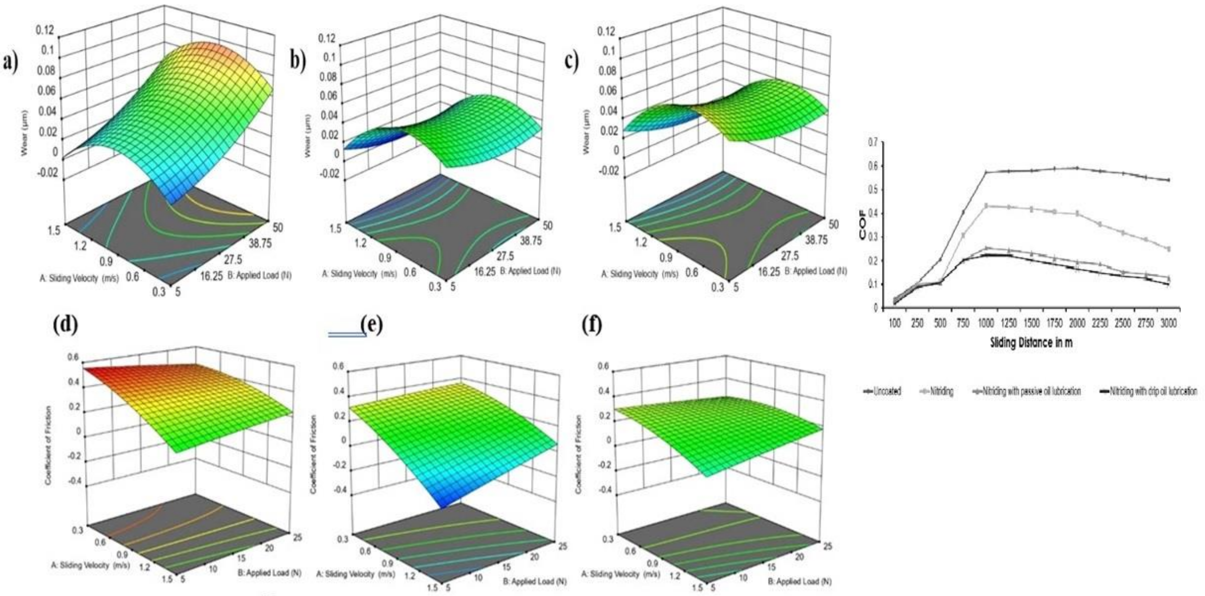

a)
b)
c)
(d)
(e)
(f)
Wear (µm)
A: Sliding Velocity (m/s)
B: Applied Load (N)
Coefficient of Friction
COF
Sliding Distance in m
Uncoated
Nitriding
Nitriding with passive oil lubrication
Nitriding with drip oil lubrication

Figure 5

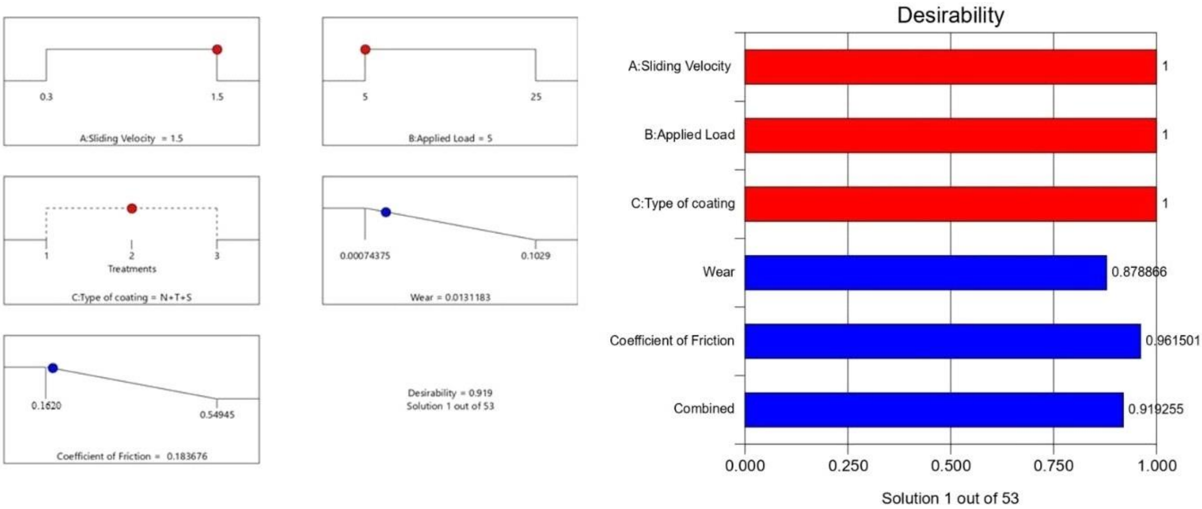

0.3
1.5
A:Sliding Velocity = 1.5
5
25
B:Applied Load = 5
1
2
3
Treatments
C:Type of coating = N+T+S
0.00074375
0.1029
Wear = 0.0131183
0.1620
0.54945
Coefficient of Friction = 0.183676
Desirability = 0.919
Solution 1 out of 53
Desirability
A:Sliding Velocity
1
B:Applied Load
1
C:Type of coating
1
Wear
0.878866
Coefficient of Friction
0.961501
Combined
0.919255
0.000
0.250
0.500
0.750
1.000
Solution 1 out of 53

Figure 6