

Influence of Parameters on Sliding Wear of Titanium Nitride Coated 6061 Aluminium Alloy

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ABSTRACT

The objective was to study the wear behaviour of titanium nitride deposited onto 6061 aluminium alloy by DC magnetron sputtering technique. The coating on the aluminium substrate was characterized using optical microscopy, scanning electron microscopy and energy dispersive spectroscopy analysis. The hardness of the uncoated and coated aluminium specimens was tested using Vickers hardness tester and the results revealed that coated specimens had 26 % improvement compared to uncoated samples. The dry sliding wear behaviour of the titanium nitride coated aluminium specimens was investigated using pin on disc tribometer by response surface methodology. Five level central composite design was utilized and experiments were conducted by varying the wear process parameters such as load, velocity and sliding distance within the range 15 to 45 N, 1.5 to 4.5 m/s and 500 to 1500 m respectively. Wear test results revealed that increase in load increases the wear rate and increase in velocity and sliding distance decreases the wear rate. The developed model was validated through the comparison of experimental and model values which yields an error within 3 % confirms that the model was adequate. Scanning electron microscope was utilized to analyze the worn surfaces to study the wear mechanisms.

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1. INTRODUCTION

Aluminium alloys are largely applied in various fields due to its inherent benefits like high strength to weight ratio, low density, better formability and high thermal conductivity. But the aluminium alloys are limited in the wear applications due to its disadvantages such as less hardness and poor wear resistance [1]. Enhancement of wear resistance of aluminium alloys through reinforcing

by hard ceramic particles is one of the beneficial methods, but the level of wear property improvement is less [2]. Particle reinforced aluminium composites have been numerously fabricated and investigated for their properties [3-4]. The greater improvement of wear resistance of aluminium alloy will certainly make an impact on the weight critical applications where high wear resistance is required. The promising and innovative methodology for such improvement is

the coating technology because these coating films provide better hardness, good wear resistance and less friction, which noticeably increases the lifetime of the components [5]. A large number of coatings and their deposition techniques are available for the modification and protection of the surface of the materials. The recently developing surface modification methods to improve the life time of the materials are chemical vapor deposition (CVD) and physical vapor deposition (PVD) among the various techniques available in the industry [6].

Magnetron sputtering technique under PVD has been under use for the important industrial coating applications in the recent years due to its outstanding performance by providing harder, less friction and high wear resistant coating when compared to other sputtering techniques [7]. A review has been made on the various PVD techniques available for hard coating and it is reported that controlling of the process parameters will help in achieving the desirable properties for the specific application [8]. Effect of PVD process parameters such as bias voltage, etching and coating time on the hardness of AlMgSi1 alloy is investigated and it is concluded that optimum controlling of process parameters resulted in 15 % increase of hardness in the coated aluminium alloy [9]. Coating has been made on magnesium alloy through magnetron sputtering technique and hardness has been tested using universal hardness equipment. The results revealed that there is hardness improvement of about 70 % in the coated alloy compared to uncoated alloy and also it is reported that some defects have been observed on the coated layers [10].

Investigation on the tribological behaviour of the coating on the metal substrate is an essential thing to make use of these surface engineering methods in various applications which avoids the degradation of the materials in the form of wear. These ceramic coating of few micrometers on the substrates provides high wear resistance performance and thus it can be well effectively applicable for tribological applications like cylinder liners, pistons and brake drum. There are various statistical techniques available for study of tribological behaviour such as Taguchi's method, response surface methodology (RSM) and artificial neural network (ANN) [11-13]. Among these techniques, RSM is most commonly

in use for the engineering applications and in the industrial field where the performance of a product is influenced by several factors [14]. Sliding wear behaviour of aluminium alloy and aluminium hybrid composite is investigated using RSM technique under the influence of load, speed and sliding distance. The RSM results revealed that load contributes much on wear rate of the specimens followed by speed and distance [15]. Al 6061/silicon carbide (SiC) composite fabricated using stir casting technique has been studied for its wear characteristics and optimization of the parameters to minimize the wear rate has been done using RSM, and the optimization results reveals that a desirability of 87.75 % can be attained [16].

From the above survey, it is understood that the wear behaviour of the coating on aluminium 6061 substrate is not explored hence this study deals with coating of titanium nitride (TiN) over the aluminium alloy and investigation of its dry sliding wear behaviour using RSM technique.

2. MATERIAL SELECTION

Al6061 alloy is chosen as the substrate due to its enlarged usage in automotive sector particularly for the development of engines block, body panels and chassis. Ti made sputtering target is preferred for coating on the substrate due to its high melting point and better hardness. The elemental analysis of the Al6061 aluminium alloy is given in Table 1.

Table 1. Elemental analysis of Al6061 alloy.

Elements	Composition (%)
Al	98.18
Si	0.312
Fe	0.172
Cu	0.303
Mn	0.159
Mg	0.828
Others (Cr, Ni, Zn, Sn, Ti, Pb, Ca)	0.048

2.1 Experimental Procedure

Aluminium 6061 is coated with TiN through DC magnetron sputtering technique. The specimens are machined to the size of 8 x 8 mm and ultrasonically cleaned using acetone and diluted HCl. Then the specimens are dried using the

nitrogen gas and kept in the deposition chamber. The sputtering target made of Ti is used for deposition and argon gas is used as a carrier gas for deposition of TiN over the substrate. The base pressure of 5×10^{-6} mbar is developed in the chamber and operating pressure of 8×10^{-4} mbar is maintained within the chamber. The aluminium alloy substrates are preheated to the temperature of 250 °C and the deposition is carried out for 3000 seconds on all the specimens.

2.2 Hardness evaluation

The specimens are machined out from the uncoated aluminium alloy and the coated aluminium alloy for its evaluation of micro hardness. The specimens are polished by emery sheets to achieve smooth surface. Then the specimen is fixed over the specimen plate by the holding jaws. The diamond indenter in the Vickers Hardness tester is employed to make indentation on the surface of the specimens and the load of 100 gf is applied for the time of 15 seconds. The diagonal measurement of the indentation gives the hardness value at the tested spot. The hardness of the surface is tested for three times on both coated and uncoated aluminium alloy and the average value is taken. The obtained hardness values for the uncoated and coated aluminium alloy are 88 HV and 119 HV respectively. The hardness is found improved 26 % in the coated specimens compared to the uncoated aluminium specimens. This is due to the harder TiN coating on the substrate which has the high capability of load bearing leads to reduction in the level of indentation produced by the indenter. The uncoated specimens has the softer surface which has poor load bearing capability and the action of indenter during the hardness test produces high deformation on the surface and resulted in less hardness.

2.3 Characterization of TiN coating

The microstructural examination of the coated and uncoated specimens is carried out using Zeiss Axiovert 25 CA Inverted Metallurgical Microscope (optical). The uncoated aluminium specimens are polished using linisher polisher initially and followed by emery sheets. Then the surface is polished by using disc polisher. Finally, keller's reagent is used for etching the specimens prior to surface examination. The observed micrographs of the specimens are

shown in Figs. 1a and 1b. The microstructure of the coated specimen (Fig. 1b) ensured the presence of TiN by revealing its golden bronze colour. The cross sectional view of the coating has been examined using the scanning electron microscope (SEM) and shown in Fig. 2a. The clear transition is present between the aluminium substrate and the TiN coating. There is no delamination or cracks present at the coating and substrate interface which reveals that there is good adherence is attained in the coating process. The energy dispersive spectroscopy analysis (EDS) is carried out on the TiN coated surface to analyze the presence of various elements and the obtained spectrum is shown in Fig. 2b. The peaks in the graph indicate the level of presence of the particular element and it is observed that coated surface has the major presence of titanium (Ti).

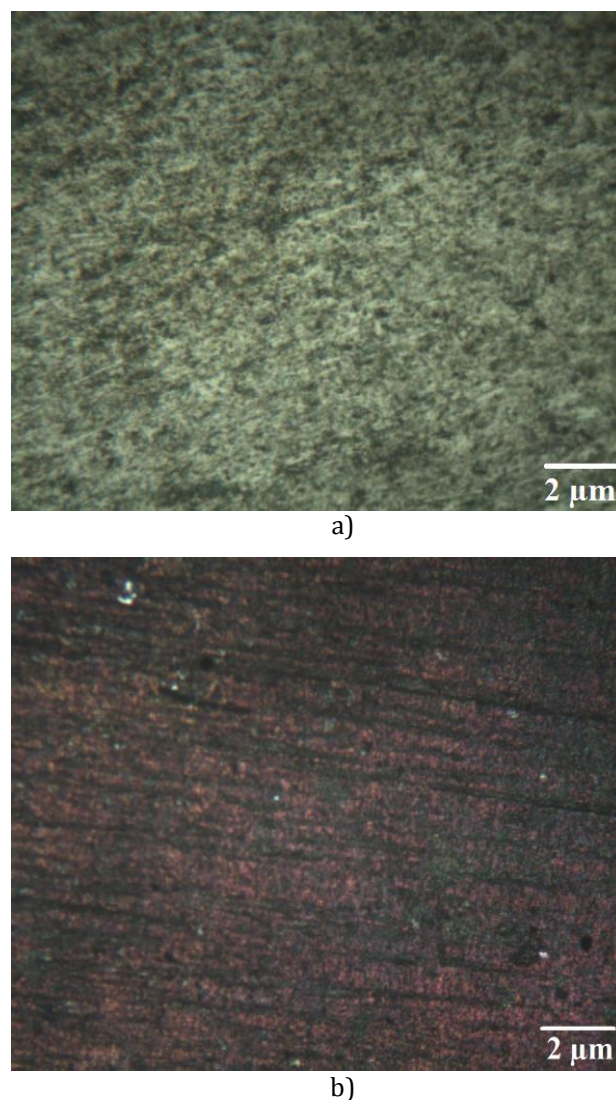


Fig. 1. Microstructures of the a) uncoated and b) coated aluminium specimens.

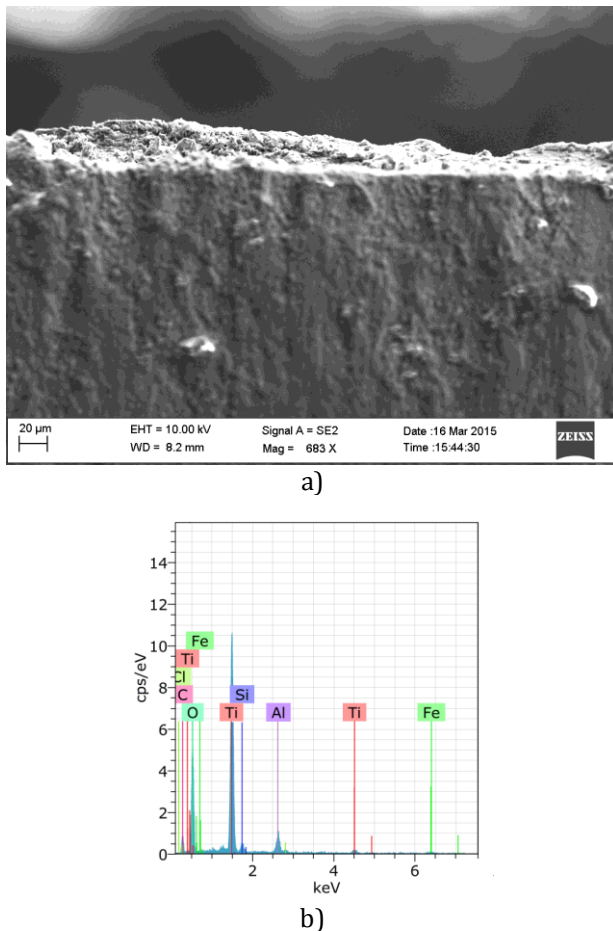


Fig. 2. a) Cross section of TiN coating and b) EDS spectrum of TiN coated surface.

2.4 Response Surface Methodology

RSM is a statistical and mathematical way of technique which is used for development of a product or process and it has been implemented in the conditions where different parameter affects the response. It provides a clear relationship between the parameters and the response which helps to optimize the parameters to achieve the desired response. The RSM comprises of planning of experiments, determination of model and checking adequacy of developed model.

2.5 Design of Experiments

The parameters selected for studying the dry sliding wear behaviour of the coated aluminium specimens are load, velocity and sliding distance. Defining of limits of the selected parameters is an important step in the RSM technique. The wide range of each parameter is selected based upon the capability of the pin on disc tribometer to obtain clear study of the response. The

relationship between the parameters and the response is given in the form of second order polynomial model, which is shown in Eqn. (1):

$$Y_u = b + \sum b_i x_{iu} + \sum b_{ii} x_{iu}^2 + \sum b_{ij} x_{iu} x_{ju} \quad (1)$$

Where Y_u is the response and the coefficients are b_i , b_{ii} and b_{ij} . Second term in the Eqn. (1) denotes the linear effect, third term denotes the higher order effect and the last term denotes the interaction effect.

Table 2. Process parameters with their levels.

Factors	Levels				
Load (N)	15	21	30	38	45
Velocity (m/s)	1.5	2.1	3	3.8	4.5
Sliding distance (m)	500	702	1000	1297	1500

Planning of experiments is done through central composite design (CCD) using statistical software. The 20 experimental runs are generated for the selected number of parameters. An alpha value of 1.682 is selected and hence the parameters are varied for five levels which are shown in Table 2.

2.6 Dry Sliding Wear Test

The dry sliding wear behaviour is studied on the coated Al6061 specimens to study the effect of TiN coating on the improvement of wear resistance. The coated specimen is fixed in the specimen holder of the pin on disc tribometer and the load is applied through the lever mechanism on the specimen which makes the specimen to well adhere to the counterface. The counterface is made of steel with hardness 65 HRC and the specimen is kept in contact to this counter face at the constant track diameter of 90 mm. The disc is polished regularly with SiC emery sheets for fresh surface contact with the coated specimen surface. The parameters are set in the tribometer setup and experimental runs are performed as per the obtained experimental design generated through RSM technique. The wear rate of the specimens is calculated through the weight loss method (Eqn. (2)) by weighing the specimen before and after the sliding wear test.

$$W = \frac{M}{\rho D} \quad (2)$$

Where 'W' is the wear rate (mm^3/m), 'M' is weight loss (g), 'p' is density (g/mm^3) and 'D' is the sliding distance (m).

3. RESULTS AND DISCUSSIONS

The effect of wear process parameters on the TiN coated aluminium substrate is investigated through RSM technique and presented in the following sections briefly.

3.1 Response Surface Model

The sliding wear test results obtained for 20 set of experiments conducted as per experimental design are shown in Table 3.

Table 3. Experimental wear rate values of coated aluminium specimens.

S. No.	Load (N)	Velocity (m/s)	Sliding distance (m)	Wear rate (mm^3/m)
1	30	3.0	500	0.00371
2	30	3.0	1500	0.00276
3	30	1.5	1000	0.00399
4	30	3.0	1000	0.00349
5	38	3.8	1297	0.00389
6	30	3.0	1000	0.00331
7	15	3.0	1000	0.00102
8	30	4.5	1000	0.00302
9	30	3.0	1000	0.00358
10	45	3.0	1000	0.00586
11	38	2.1	1297	0.00422
12	21	2.1	702	0.00295
13	30	3.0	1000	0.00367
14	21	3.8	1297	0.00210
15	38	3.8	702	0.00452
16	38	2.1	702	0.00492
17	21	2.1	1297	0.00236
18	30	3.0	1000	0.00389
19	21	3.8	702	0.00237
20	30	3.0	1000	0.00314

The experimental results are analyzed through the statistical software and the model has been developed to predict the response. The significance test is conducted to estimate the coefficients for the development of model and shown in Table 4.

Using the regression coefficients from Table 4, sliding wear rate is expressed in the form of Eqn. (3):

$$\text{Wear rate} = 0.000134 + 0.000162 L - 0.000524 V + 0.000001 D + 0.000004 V*V + 0.000002 L*V \quad (3)$$

where L is load (N), V is velocity (m/s) and D is distance (m).

Table 4. Significant test results for wear rate.

Term	Coef	SE Coef	T	P
Constant	0.000134	2.96E-3	0.045	0.965
Load (N)	0.000162	9.2E-5	1.763	0.108
Velocity (m/s)	-0.000524	9.21E-4	-0.570	0.582
Sliding distance (m)	0.000001	3.0E-6	0.457	0.657
Load (N) * Load (N)	-0.000000	1.0E-6	-0.222	0.829
Velocity (m/s) * Velocity (m/s)	0.000004	1.11E-4	-0.039	0.97
Sliding distance (m) * Sliding distance (m)	-0.000000	0.000	-1.046	0.32
Load (N) * Velocity (m/s)	0.000002	1.5E-5	0.116	0.91
Load (N) * Sliding distance (m)	-0.000000	0.000	-0.497	0.63
Velocity (m/s) * Sliding distance (m)	0.000000	0.000	0.413	0.639

Table 5. Comparison of the model and experimental values.

S. No	L (N)	V (m/s)	SD (m)	Exp wear rate (mm^3/m)	Model wear rate (mm^3/m)	Error (%)
1	18	1.8	600	0.00285	0.00278	2.4
2	25	2.5	900	0.00404	0.00392	2.9
3	35	3.5	1100	0.00544	0.00536	1.4

The R^2 and adjusted R^2 values obtained for the significance test are 94.8 % and 90.2 % respectively which evident that the developed model relates the process parameters well with the response. The new levels of parameters are substituted in the above model and compared the model values with the obtained experimental values (Table 5). The error difference between these two values falls within 3 % which ensures that the model is well adequate in relating the parameters with the wear behaviour of coated aluminium alloy.

3.2 Analysis of Variance

The analysis of variance (ANOVA) is conducted for the confidence level of 95 % and significance

level of 5 % and shown in Table 6. Through this analysis, the significance of the regression model, linear, square and interaction terms as well the lack of fit can be identified. The significance of the term is decided by the P-value. If the term has lower P-value, it has greater significance over the response. From the results, it is identified that regression model has the highest significance ($P=0.000$) on the wear rate followed by linear term ($P=0.282$), square term ($P=0.771$) and interaction term ($P=0.931$). The model accuracy is checked through the significance of lack of fit. The model has the standard F value of 5.05 and it is known that if the F value of lack of fit is less than the standard F value, the model is found adequate. The lack of fit has the F value of 2.17 which confirms that the developed model is adequate.

Table 6. ANOVA for wear rate.

Source	DF	F	P
Regression	9	20.4	0.000
Linear	3	1.47	0.282
Square	3	0.38	0.771
Interaction	3	0.14	0.931
Residual Error	10		
Lack-of-Fit	5	2.17	0.208
Pure Error	5		
Total	19		

3.3 Influence of Process Parameters on Wear Rate

The plot generated for wear rate with respect to variation in parameters (load, velocity and sliding distance) is shown in Fig. 3a-3c. The variation of wear rate obtained for change in load is shown in Fig. 3a. From the plot, it is observed that the wear rate gets increased with increase in load. At low load of 15 N, smaller pressure acts through the lever arm and resulted in smaller wear rate.

The increase in load increases the wear rate due to higher contact stress that takes place on the tribo system. When the load is turned higher to 45 N, more stress acts on the counter face through the specimen which produces more removal of material and the same mechanism is attained [17]. But the presence of TiN coating on the substrate reduces the severity level of deformation and the amount of material removal due to its good adherence, stability and high wear

resistance. Therefore, this high wear resistance coating provides better tribological properties to the aluminium substrate which meets the performance requirement in the applications where high load sliding contact exists.

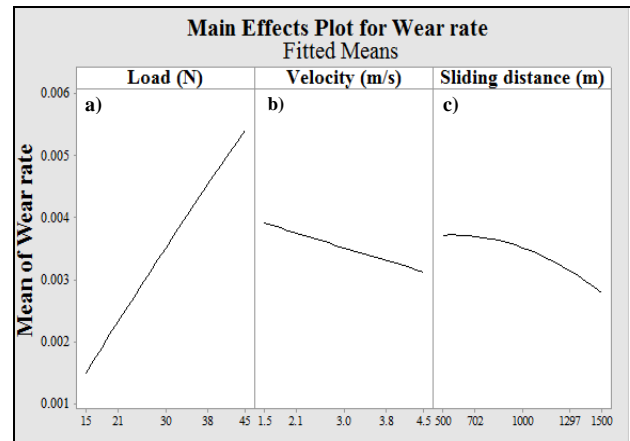


Fig. 3. Plot for wear rate with respect to a) Load b) Velocity and c) Sliding distance.

The plot for wear rate with respect to the varying velocity of 1.5 m/s to 4.5 m/s is shown in Fig. 3b. At low velocity of 1.5 m/s, the wear rate is found high as this is owing to more interaction time of the counter face steel disc with the specimens. The release of fragments of coating occurs at this stage and resulted in high wear rate. On increasing of the velocity, wear rate gets decreases and this is due to the increase in temperature at the interface causes the transformation of materials between the two interacting surfaces. This transferred material produces a mechanically alloy layer over the surface of the specimen which diminishes the direct contact between the surfaces.

This action prevents the adhesion wear in the course of sliding of the specimen with the counter face, thereby it leads to less wear rate and the same mechanism is witnessed [18].

The variation of wear rate for sliding distance is shown in Fig. 3c. At initial sliding, the wear rate is found high and this might be due to irregularities caused during the coating process. These irregularities on the coating surfaces depend upon various influencing factors during the deposition and also on surface quality of the substrates. Therefore this surface makes non uniform contact with the steel disc at the initial stage and more material gets removed from the surface. On increase of the sliding distance, the

coated surface irregularities gets removed away and makes uniform contact with the counter face and results in reduced wear rate. Thus, it is understood that increase in sliding distance reduces the sliding wear rate.

3.4 Response Optimization

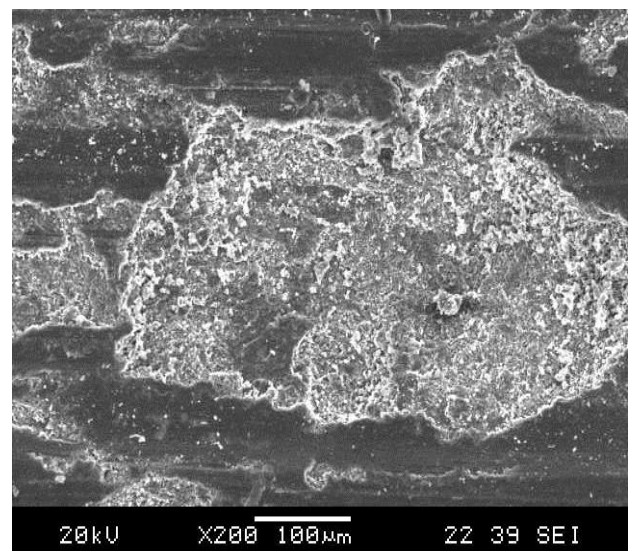
Optimization is carried out in RSM by relating the wear process parameters such as load, velocity and sliding distance with the sliding wear behaviour. The objective of this optimization process is to minimize the sliding wear rate of the coated aluminium specimens. The inputs given for the process are upper limit and the target of wear rate to obtain the optimal solution. The global solution obtained for achieving the wear rate of $0.00102 \text{ mm}^3/\text{m}$ is load of 15 N, velocity of 4.5 m/s and sliding distance of 569 m.

3.5. Scanning Electron Microscopy Analysis

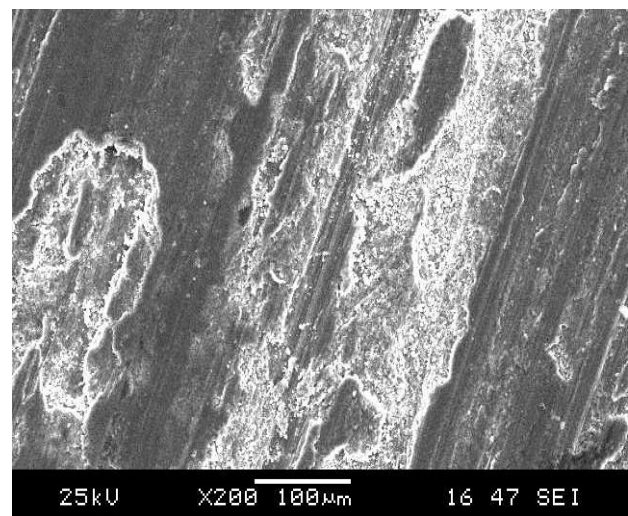
The worn out surfaces of the coated aluminium specimens are examined using SEM and the obtained micrographs are shown in Figs. 4a–4g. The coated aluminium surfaces worn at different load is considered for analysis and this helps to control the parameter which affects the wear behaviour. The surface worn at low load of 15 N (Fig. 4a) shows smaller number of scratches with mild wear due to low pressure exerted on the steel disc. This is due to the wear resistance provided by the hard coating which results in less material removal. The transition of load to 30 N (Fig. 4b) on the surface experiences more pressure at the interface which produces little removal of the hard TiN coating and is observed as the grooves on the surface. These small fragments of the coating material traps between the specimen surface and the counterface during sliding and on further sliding with more pressure at this load condition, produces continuous grooves on the specimen surface which increases the wear rate. At high load of 45 N (Fig. 4c), the worn out surface reveals more material removal in the form of debris as this is due to higher contact stresses at the interface which results in severe wear. Thus it is interpreted that on increase of load the wear gets transitioned from the stage of mild adhesion wear to severe wear.

Figure 4d and 4e shows the wear mechanisms occurred during the change of velocity from 1.5 m/s to 4.5 m/s on the surface of the specimen.

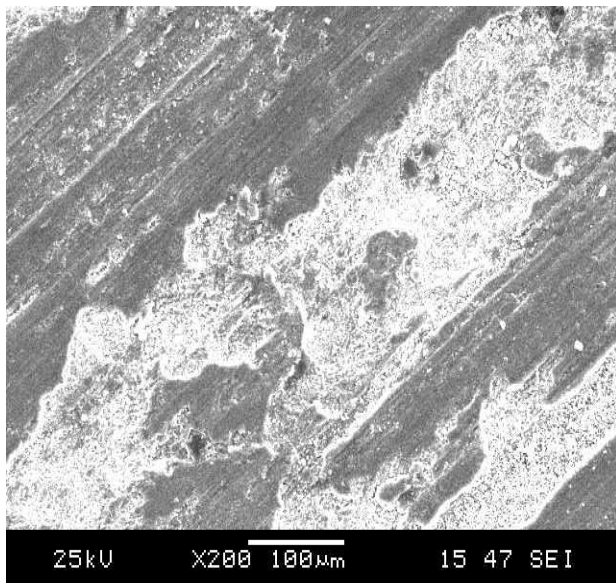
The surface worn at velocity of 1.5 m/s (Fig. 4d) shows more grooves on the surface as this is owing to more contact time at low velocity. When the velocity turned high (4.5 m/s), better wear resistance is observed as the worn out surfaces (Fig. 4e) reveals the presence of only few scratches on the surface due to deficient in interaction time. Figure 4g shows the surface worn at sliding distance of 1500 m which reveals the micro grooves on the surface with less material removal as compared to the surface worn at sliding distance of 500 m (Fig. 4f). As this is due to the absence of irregularities on the surface at the sliding distance of 1500 m which provides smooth contact action of the specimen with the counter face. From the SEM analysis (Fig. 4a–g), it is observed that worn out surfaces at all conditions show less severity with less surface damage as this is due to the presence of hard TiN coating on the aluminium substrate.



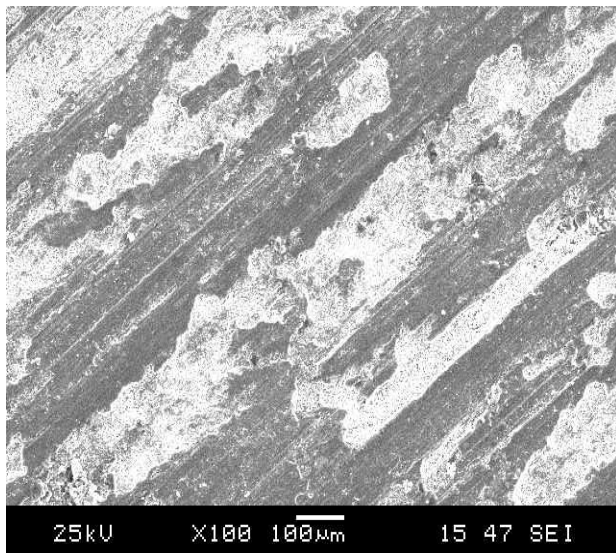
(a)



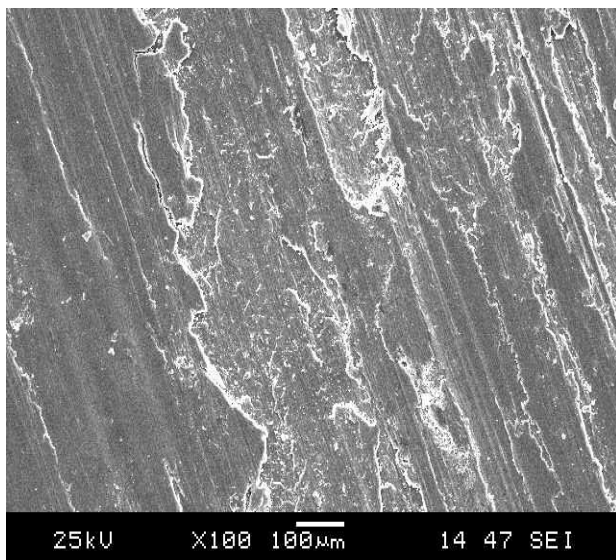
(b)



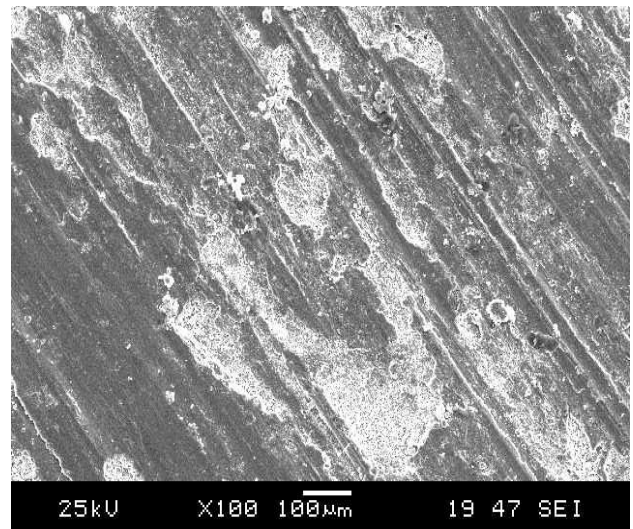
(c)



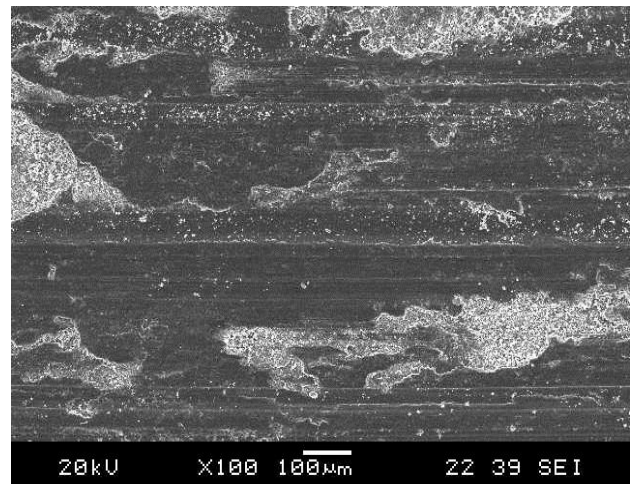
(d)



(e)



(f)



(g)

Fig. 4. SEM micrographs of worn-out surfaces at (a) $L = 15$ N, $V = 3$ m/s, $D = 1000$ m, (b) $L = 30$ N, $V = 3$ m/s, $D = 1000$ m, (c) $L = 45$ N, $V = 3$ m/s, $D = 1000$ m, (d) $L = 30$ N, $V = 1.5$ m/s, $D = 1000$ m, (e) $L = 30$ N, $V = 4.5$ m/s, $D = 1000$ m, (f) $L = 30$ N, $V = 3$ m/s, $D = 500$ m, (g) $L = 30$ N, $V = 3$ m/s, $D = 1500$ m.

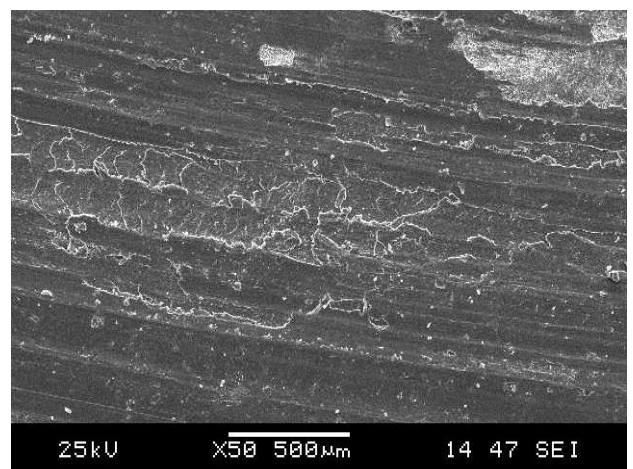


Fig. 5. SEM micrograph at optimum condition.

The optimal solution resulted through the RSM optimizer is also taken for SEM examination to check the adequacy of the developed model. The surface (Fig. 5) worn at load of 15 N, velocity of 4.5 m/s and sliding distance of 569 m reveals fine scratches along with shallow grooves on the surface. This evident that this model is good in relating the wear behaviour of the coated aluminium specimens with the dry sliding wear process parameters.

4. CONCLUSION

Titanium nitride is deposited on the aluminium substrate successfully through the DC magnetron sputtering technique. The presence of coating has been ensured through optical microscopy and scanning electron microscopy analysis. The elemental analysis shows presence of titanium content on the aluminium substrate. The hardness of the coated aluminium specimens is increased 26 % compared to uncoated aluminium alloy. Response surface methodology develops a model that provides a better relationship of wear process parameters with the sliding wear behaviour of the coated aluminium specimens. Analysis of variance results shows that the model is well adequate with less lack of fit. Wear plots interprets that increase in load increases the wear rate and increase in velocity and sliding distance decreases the wear rate. Based on the results obtained in wear test, the optimum parametric condition found out through the optimization process (in Minitab) to achieve minimum wear rate is load of 15 N, velocity of 4.5 m/s and sliding distance of 569 m. It is observed that the tribological properties of the aluminium substrate is significantly improved through the coating of titanium nitride and this surface engineered material acts as potentially wear resistant materials for the replacement of the conventional made aluminium components which lacks in their wear resistance.

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