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Optimization of wet sliding wear parameters of Titanium grade 2 and grade 5 bioimplant materials for orthopedic application using Taguchi method

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Abstract

In this research ball on disc wear tests have been carried out on Titanium grade 2 and Titanium grade 5 material using ASTM G-99 standard at room temperature in simulated body fluid. The tribological property such as the weight loss was studied by using the Taguchi design of experiments. The design of experiment was done using an L18 orthogonal array. An analysis of variance demonstrated that the individual contribution such as the applied normal load factor was 91.17% for weight loss, which means it is highest individual contribution as compared to other factors so it was concluded that weight loss is mainly influenced by the applied normal load. The analysis of the signal to noise ratio shows that the optimal weight loss was obtained with titanium grade 5 material at an applied normal load of 5 N with sliding velocity 0.05 m/s for sliding distance of 100 m. The results showed that weight loss increases with increasing the applied normal load and sliding velocity. The microstructure of all substrate materials was analyzed using a scanning electron microscope. Wear track study showed that adhesive dominant wear mechanism for both the grades.

1. Introduction

For improvement of research in the field of biomaterials engineering, it is necessary to study the tribological behavior of the material to improve the existing used biomaterials otherwise for the development of new virgin materials with exceptional properties [1]. Generally, the metal alloy has been used largely in the manufacturing of orthopedic implants so the highest quality and top superiority of orthopedic implants and instrumentation along with feasible cost are the demand of the 21st-century world. For safe and effective use of orthopedic implants that are left in vivo for a long period, it is necessary to study the tribological behavior of orthopedic bioimplant substrate materials. Wear is explained as the "loss of material in particulate form as a consequence of relative motion between two surfaces" [2]. Several factors contribute to the long term survivorship in vivo of a biomedical implant. Out of this wear can be a major influencing factor for the proper performance of orthopedic bioimplant materials in an actual condition [3]. Bone and bone tissue suffer substantial loads during physical activity of the human body so it is mandatory to have a better load-bearing characteristic of artificial implants. Load bearing orthopedic bioimplants material like knee and hip joints are made from stainless steels and titanium alloys because of their better mechanical and corrosion properties [4,5]. Ti-based alloys are an important bioimplant materials generally used in total hip joint replacement. This alloys when compared with Co–28Cr–6Mo exhibits more wear which is primarily due to abrasion and cracking [6]. For better durability in the human body, the superior wear mechanism and surface modification process was suggested for orthopedic implant material for long life and proper work functionality [7]. Generally, the modes of failure in engineering materials are corrosion failure, fatigue failure and wear failure and wear failure is more prevailing in joint prostheses [8,9]. The tribological properties of material like wear rate, wear mechanism largely depends upon the manufacturing process of alloys. The heat treatment methods have more influence on the performance of bioimplant alloys [10]. So for the effectiveness of bioimplant material in the living body, it is necessary to study all aspects of bioimplant material. High and considerable wear rate is a serious problem in the orthopedic implant. Wearing or surface rubbing between two parts leads to the production of the metal ions. These ions contact with blood and tissues which causes a serious problem. So to avoid this it is required to impart superior properties to the material [11]. Nickel-Titanium (NiTi) alloy is the most elastic and bio-compatible alloy compared with other Ni alloys. For more superior properties of NiTi material, Graphene Oxide/Silver nanoparticle nanocomposite coating is a promising coating material by using the electrophoretic deposition technique to improve the mechanical properties and friction coefficient of NiTi alloy [12]. There are various composite fabrication techniques like laser surface alloying, plasma spraying, electrophoretic deposition, aerosol deposition, etc. Out of this Plasma spraying is a promising technique. Uncoated 30% glass fiber filled Polyetheretherketone (PEEK-30% GF) has better wear characteristics as compared to Ti-6Al-4V, SS316L, PEEK (general) and

Polyurethane so uncoated 30% glass filled polyether-etherketone can be used as alternative material for human orthopedic implants. However, PEEK-30% GF has lower biocompatibility as compared to other materials which is a fundamental requirement for biomaterials to function adequately [13]. Babu et al. [14] focusses on the tribological properties of Ti-3Al-2.5V titanium alloy by using the Taguchi method. Results revealed that the optimum process parameters can be found using Taguchi method based on the regression equation. Kokubo's recipe is used to prepare the simulated body fluid (SBF). This solution contains various ions and has a common pH of 7.25 similar to human plasma [15]. The chemical composition is given below in Table 1.

Chemical composition and density of Titanium grade 2 (TIGR2) and Titanium grade 5 (TIGR5) material are shown in Table 2.

Table 1. Chemical ingredients of SBF.

Ingredient of SBF	Amount ($g \cdot l^{-1}$)
$NaCl$	7.995
$NaHCO_3$	0.351
KCl	0.223
$K_2HPO_4.3H_2O$	0.229
$MgCl_2 \cdot 6H_2O$	0.304
$CaCl_2$	0.279
Na_2SO_4	0.072
$(CH_2OH)_3 CNH_2$	6.056
1 $kmol \cdot m^{-3}$ HCI	Adjust the pH-7.25
Ultrapure water	Volume up to 1 litre

Table 2. Chemical composition (%) and density of materials ($g \cdot mm^{-3}$).

Material	TIGR2	TIGR5
N (Nitrogen)	0.02%	0.04%
C (Carbon)	0.1%	0.005%
H (Hydrogen)	0.020%	0.0125%
Fe (Iron)	0.2%	0.3%
O (Oxygen)	0.25%	0.10%
Al (Aluminum)	-	5.7%
V (Vanadium)	-	3.89%
Ti (Titanium)	99.41%	89.95%
Density ($g \cdot mm^{-3}$)	4.51×10^{-3}	4.43×10^{-3}

In the reported data there is no information available about weight loss values under wet conditions as per the process parameters or input variables. The values of input variables and their levels in the present study are given in Table 3. In the available literature, there are no detailed findings on tribological behavior such as weight loss of TIGR5 and TIGR2 materials by using the statistical approach. So we used Taguchi experimental design method in the present work.

Table 3. Input variables and their levels.

Name	Level values	Column	Level
Type of Material	TIGR5, TIGR2	1	2
Load (P) in N	5, 10, 20	2	3
Velocity (V) in $m \cdot s^{-1}$	0.05, 0.1, 0.2	3	3
Sliding Distance (SD) in m	100, 200, 300	4	3

2. Materials and method

Wear ball on disk machine (DUCOM, Bangalore, India) was used to study the weight loss of TIGR5 and TIGR2 materials. Specimen of 40 mm diameter and 6 mm thickness TIGR5 and TIGR2 material (Substrate) were polished to a roughness value of 0.2 to 0.5 μm. A steel ball (indenter) of 10 mm diameter was used. The wear between the specimen and steel ball was noted by the linear variable differential transformer (LVDT) sensor which was mounted on the machine. The TIGR5 and TIGR2 were tested under wet conditions at different applied normal load (P), Sliding velocity (V) and Sliding distance (SD) as per the ASTM G-99 standard. For wet condition, the specimen was immersed in SBF using the same test condition. Load bearing orthopedic bioimplant material such as total hip joint has three components. They are referred as stem, femoral ball hip and the acetabular cup. These components are mechanically integrated to get the required compactness. The femoral ball is in direct contact with acetabular cup so femoral ball experiences friction and wear because of articulating contact. The wear parameters for the present study are selected based on the wear conditions faced by the femoral head inside the acetabular cup. The stress induced in a hip joint during walking is in the range of 0.8-2.5 MPa [16]. Hip joint is expected to withstand high frictional forces for a minimum of 20 years [17]. Since wear experiments cannot be continued for such a long period, the normal load during the test is kept at a high value of 5 N, 10 N and 20 N, to obtain significant wear loss data.

The Pin on disc tests were carried out on TIGR5 and TIGR2 substrate materials by taking the Taguchi design (Mixed Level design) of experiments. To accomplish the objective of the experimental study in the present work, four different parameters were used as the input source. The experimental levels were decided with the support of the L18 orthogonal array. Taguchi analysis was carried out with open source software Minitab 17.

3. Result and discussion

Before the start and end of every test, the weight of substrate materials was noted by the Electronic weighing machine (Contech Instruments Ltd., Navi Mumbai, India). The design layout matrix using L18 orthogonal array and experimental results are shown in Table 4.

The values of coefficient of friction (COF) are obtained from the data recorded in the form of an excel sheet from the WINDUCOM 2010 machine software. Wear ball on disk machine is attached to a computer (ACER VERITON M200-H81 DESKTOP, PCI-E -6321 NI Card, DUCOM, Bangalore, India) displays all the results in graphical form. By using the design layout matrix as given in Table 4, the values of COF for TIGR5 and TIGR2 were found in the range of 0.39 to 0.54 and 0.46 to 0.63 respectively. The average value of COF are given in Table 5. These values are related to the wet test using the mentioned design layout matrix in Table 4.

Figure 1 shows bar chart of average value of COF for TIGR5 and TIGR2 material under wet condition. It is observed that COF for TIGR5 material is found to be less as compare to TIGR2 material.

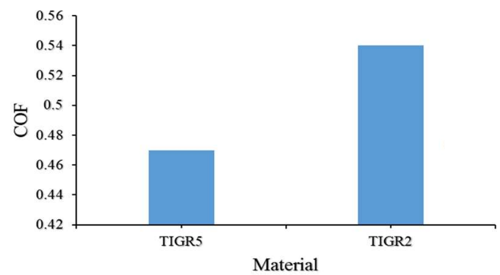

Figure 1. Average value of COF for TIGR5 and TIGR2.

Table 4. Design layout matrix and experimental results.

Experimental No.	Type of material	Load (P)	Velocity (V)	Sliding distance (SD)	Weight loss in gram	S/N Ratio (dB)
1	TIGR5	5	0.05	100	0.0031	49.7522
2	TIGR5	5	0.10	200	0.0036	48.7215
3	TIGR5	5	0.20	300	0.0038	48.2197
4	TIGR5	10	0.05	100	0.0052	45.3912
5	TIGR5	10	0.10	200	0.0060	44.3606
6	TIGR5	10	0.20	300	0.0063	43.8587
7	TIGR5	20	0.05	200	0.0095	40.9675
8	TIGR5	20	0.10	300	0.0096	40.6163
9	TIGR5	20	0.20	100	0.0105	40.0697
10	TIGR2	5	0.05	300	0.0042	47.7408
11	TIGR2	5	0.01	100	0.0045	47.3448
12	TIGR2	5	0.20	200	0.0050	46.1634
13	TIGR2	10	0.05	200	0.0071	43.1384
14	TIGR2	10	0.10	300	0.0073	42.7872
15	TIGR2	10	0.20	100	0.0080	42.2406
16	TIGR2	20	0.05	300	0.0105	39.3942
17	TIGR2	20	0.10	100	0.0106	38.9982
18	TIGR2	20	0.20	200	0.0120	37.8168

Table 5. Average value of COF during wet test.

Materials	Range of values for COF obtained	Average of COF
TIGR5	0.39 to 0.54	0.47
TIGR2	0.46 to 0.63	0.54

3.1 Signal to noise (S/N) ratio and analysis of variance (ANOVA)

The maximization of S/N ratio signifies the minimization of weight loss. The significance of the controllable factor is investigated using the S/N ratio approach and same is expressed as:

$$S/N = -10 \times \log\left(\frac{\sum Y^2}{n}\right) \quad (1)$$

Here, Y is the experimental data and n is the number of experiments. The S/N ratio values are also tabulated to find the optimal value for weight loss based on ranking. The response Table for S/N ratio for the smaller is better characteristic shown in Table 6. The main plot for S/N ratios for weight loss is shown in Figure 2.

The response Table for S/N ratios for weight loss shows the optimal value of input factors for the lowest weight loss. The bolded values in Table 6 show the optimal input factors for obtaining the lowest weight loss. The experimental results were analyzed with ANOVA. This analysis is carried out for a significance of level α = 0.05, i.e. for a confidence level of 95%. ANOVA analysis was performed to identify the significant input variable for weight loss of TIGR5 and TIGR2 material.

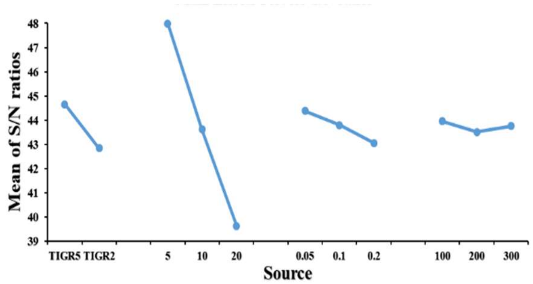

Figure 2. Main effect plot for SN ratio - Weight loss

Sources with a P-value less than 0.05 were considered to have a statistically significant contribution to the performance measures. Table 7 shows the percentage of contributions of input sources to weight loss. It was observed that applied normal load input source significantly influenced the weight loss as compared to other input sources. The percentage contribution of the applied normal load is 91.17% while type of material and sliding velocity contributed to 5.14% and 2.2% respectively. From ANOVA Table 7 it shows that the sliding distance is an insignificant input source in wear test. The error contribution is 1.47% only. It can be observed from Figure 2. the weight loss increases as the applied normal load increases in a linear fashion. The graphical representation of percentage contribution of input variables is shown in Figure 3.

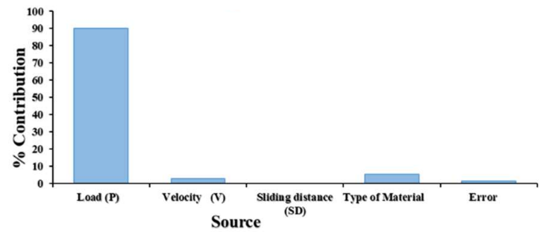

Figure 3. Percentage contribution of input variables on weight loss.

Table 6. Response Table for S/N ratio - weight loss.

Level	Type of material	Load (P)	Velocity (V)	Sliding Distance (SD)
1	44.66	47.99	44.40	43.97
2	42.85	43.63	43.80	43.53
3		39.64	43.06	43.77
Delta	1.81	8.35	1.34	0.44
Rank	2	1	3	4

Table 7. ANOVA for SN ratios - weight loss.

Source	DF	Seq SS	Adj SS	Adj MS	F-Value	P-Value	Contribution (%)
Load (P)	1	0.000124	0.000124	0.000124	930.05	0.000	91.17
Velocity V)	1	0.000003	0.000003	0.000003	23.33	0.000	2.20
Sliding Distance (SD)	1	0.000000	0.000000	0.000000	0.02	0.877	0.00
Type of Material	1	0.000007	0.000007	0.000007	56.05	0.000	5.14
Error	13	0.000002	0.000002	0.000000			1.47
Total	17	0.000136	0.000136				100
				R-squared		99.73%	
				Adjusted *R*-squared		99.34%	
				Predicted *R*-squared		98.68%	

3.2 Regression equation and model summary

The regression equation for the desired output was obtained with the help of statistical software Minitab 17. The relationship between the weight loss and the input variables were modelled for TIGR5 and TIGR2 material used in the experiments. The regression equation for the weight loss shown in Table 8.

From the model summary in Table 9, it observes that the value of R-sq is 99.73% which indicates this model can be considered for predicting the optimal process parameter combination.

Table 8. Regression Equation - weight loss.

Type of Material	
TIGR5	Weight loss = 0.000744 + 0.000421 load + 0.00667 velocity -0.000000 Sliding distance
TIGR2	Weight loss = 0.002033 + 0.000421 load + 0.00667 velocity - 0.000000 Sliding distance

Table 9. Model Summary - weight loss.

S	R-sq (%)	R-sq (adj, %)	R-sq (pred, %)
0.0003819	99.73	99.34	98.68

3.3 Verification experiment

A confirmation experiment was conducted for optimal wear parameters for TIGR5 and TIGR2 materials with the help of a response optimizer in the Minitab 17 software. The percentage of error between the experimental and predicted value at the optimal condition observed only 3.12%, 4.54% for TIGR5 and TIGR2 material respectively. This indicates predicted values are very close to the experimental values and the model is significant to predict the weight loss.

3.4 3-D surface plots for weight loss:

3-D surface plot help in the prediction of weight loss at any region of the experimental domain. Figure 4 and Figure 5 shows various 3-D surface plot for weight loss of TIGR5 and TIGR2 materials (load x sliding velocity, load x sliding distance).

From Figure 4 and Figure 5 it shows that weight loss increases with an increase in applied normal load and sliding velocity. The weight loss minutely increases with an increase in the sliding distance. With an increase in the sliding distance, the temperature between the substrate material and indenter increases due to this titanium substrate material gets oxidized, which is attributed to the low thermal conductivity of titanium alloys material. This oxidized surface of substrate material becomes stable to some extent which reduces the weight loss. The formed fragmented oxide layer or particles sometimes acts as a lubricating agent. The formation of oxide islands at some locations as evident from Scanning electron microscope (SEM). From literature, it was found that in titanium alloy at higher load and sliding distance there was the formation of oxide islands or layers [18].

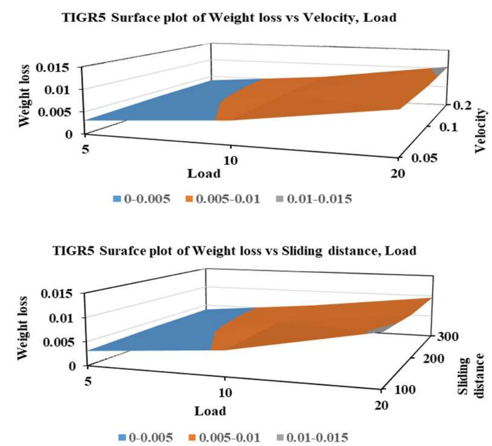

Figure 4. 3-D surface plot of TIGR5 for weight loss.

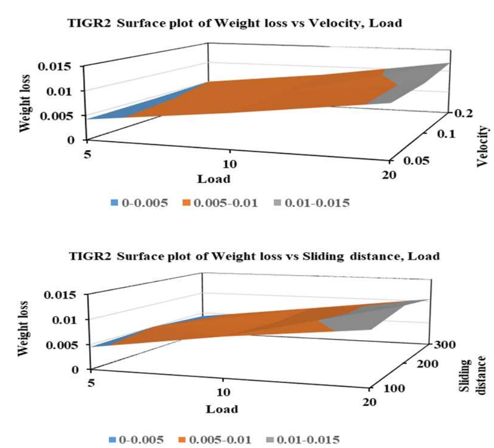

Figure 5. 3-D surface plot of TIGR2 for weight loss.

Table 10. Experimental and predicated value of weight loss in gram.

Type of material	Load (P)	Velocity (V)	Sliding distance (SD)	Predicted value of weight loss (gm)	Experimental value of weight loss (gm)	Error (%)
TIGR5	5 N	0.05 $m \cdot s^{-1}$	100	0.0032	0.0031	3.12
TIGR2	5 N	0.05 $m \cdot s^{-1}$	300	0.0044	0.0042	4.54

3.5 Microstructure study

Microstructure study of the wear tracks is carried out to analyze the wear mechanism that the TIGR5 and TIGR2 material undergo during wear testing. Figure 6 shows SEM images of TIGR5 and TIGR2 material under wet conditions. SEM images were taken at MMMF lab, IIT Bombay (Carls Zeiss Microscopy Ltd. Cambridge CB1 3JS, United Kingdom). From the SEM images of TIGR2 and TIGR5 material it is clear that TIGR2 shows more wrinkled and rough surfaces as compared to TIGR5 material. This is attributed to the hardness and wear characteristics of TIGR2 material. The lower hardness and wear resistance of the TIGR2 resulted in a more amount of wear loss as compared to TIGR5. It can be observed that the worn surface mainly consists of grooves, small cracks, partially irregular pits and very small delamination. The SEM images clearly revealed presence of fracture and chipping at some location. Mostly the dominant wear mechanisms were identified as adhesive with some traces of abrasive wear for both TIGR2 and TIGR5 material.

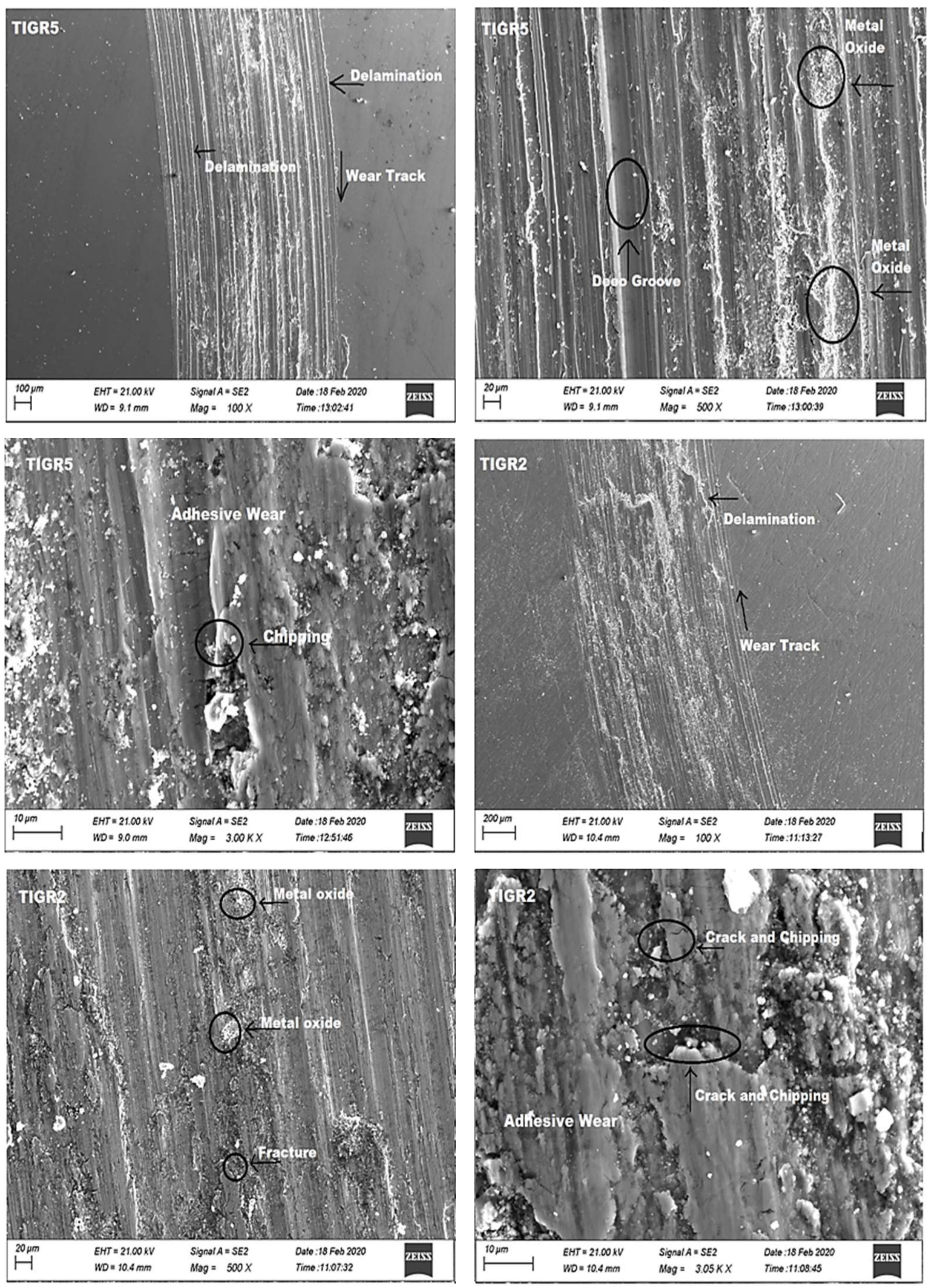

Figure 6. SEM images of TIGR5 and TIGR2.

4. Conclusions

1. Pin on disc wear test revealed that TIGR5 material exhibits lower weight loss and COF compared to other TIGR2 material.

2. From the ANOVA data, it shows that the most significant and influencing input factor affecting the weight loss is the applied normal load. For weight loss, the individual contribution of the applied normal load input factor was 91.17%.

3. The minimum value of weight loss is 0.0031 gm observed at highest S/N ratio 49.7522 dB which identify the statistically significant parameters that are an applied normal load 5 N with sliding velocity 0.05 m·s for sliding distance 100 m for TIGR5 substrate material.

4. The weight density and weight loss of TIGR2 very close to TIGR5 material observed marginally different. So as the economic and commercial availability point view, TIGR2 is the best material option for the orthopedic implant material.

5. From the SEM study, the dominant wear mechanisms were identified mostly as adhesive for TIGR5 and TIGR2 materials on the wear tracks under wet conditions.

6. For the superior performance of bioimplant material, this study demands the necessity of surface modification of various substrates by using different surface coating methods. US Food and Drug Administration (FDA) approved the Plasma spraying technique for coating implants with biomaterials. Plasma spraying is the most potential technique for synthesizing surface coating [19].

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