



Taguchi and morphological analysis on the lubricated friction and wear of titanium alloy Ti10V2Fe3Al under CNT nano-lubricant

Abdul Munir Hidayat Syah Lubis ^{1,2*}, Poppy Puspitasari ³, Avita Ayu Permanasari ³,
Muhammad Ilman Hakimi Chua Abdullah ¹, Diki Dwi Pramono ³

¹ Faculty of Mechanical Technology and Engineering, Universiti Teknikal Malaysia Melaka, Hang
Tuah Jaya, 76100 Durian Tunggal, Melaka, MALAYSIA.

² Mechanical Engineering Department, Universitas Muhammadiyah Surakarta, INDONESIA.

³ Mechanical and Industrial Engineering Department, Universitas Negeri Malang, INDONESIA.

*Corresponding author: abdulmunir@utem.edu.my

KEYWORDS

Titanium alloy
Ti-10-2-3
Friction
Wear
Taguchi
CNTs

ABSTRACT

Due to low surface oxides and plastic shearing, titanium alloys known to have low tribological properties and application of lubricants can improve these properties. This work aims to study the effect of CNT's type, load, and sliding speed on lubricated friction and wear of Titanium Alloy Ti10V2Fe3Al by Taguchi method and wear surface morphology under the CNT nano lubricants. Titanium alloy Ti 10V2Fe3Al pin was rubbed against a steel disc. Samples of nano lubricants were made from commercial oil infused with 1 ppm SWCNT's, DWCNT's, and MWCNT's. The relationship between CNT's type, testing load, and speed, to friction and wear of the Ti10V2Fe3Al is examined using Taguchi's method. The result of present work suggests that variability on friction and wear of Ti 10V2Fe3Al are most affected by the load. The SWCNT's nanolube shall provide smaller variability to friction but the MWCNT's nanolube gives smaller variability to wear. Intermediate rubbing speed gives smaller variability to friction and high rubbing speed gives smaller variability to wear. Load and speed can cause CNTs to roll, exfoliate the structure, and absorb titanium surface to form a protective mechanism. Despite presence of nano lubricant, the adhesive wear may still dominantly occur.

Received 20 August 2023; received in revised form 5 November 2023; accepted 12 January 2024.

To cite this article: Lubis et al., (2024). Taguchi and morphological analysis on the lubricated friction and wear of Titanium alloy Ti10V2Fe3Al under CNT nano-Lubricant. Jurnal Tribologi 40, pp.120-138.

1.0 INTRODUCTION

Titanium alloy is a metallic material that possesses a combination of advantageous physical, mechanical, and chemical characteristics, rendering it highly appealing for utilization in many engineering applications. Initially, titanium alloys were primarily utilized in the aerospace sector. However, advancements in material engineering have led to the inclusion of various types of these alloys in non-aerospace industries, such as biomedical, performance to sports, automotive, power generation, offshore, general engineering, and architecture. The extension of their use is attributed to the distinctive properties exhibited by titanium alloys (Dong, 2010; R.R. Boyer & Briggs, 2013). Titanium alloys possess favorable mechanical properties such as strong mechanical strength, ductility, and resistance to elevated-temperature creep (F.H. Froes, 2015). However, these alloys exhibit inadequate wear resistance and tribological properties due to their low surface oxides and susceptibility to plastic shearing (Kaur et al., 2019)

Titanium slid against steel is a typical contact pair in tribological applications which can be found in aerospace components and manufacturing process. Various works studying tribological properties of Titanium Alloy have been reported. Early investigation by Budinski (Budinski, 1991), concluded that both pure and alloy grade of titanium have poor abrasion resistance and adhesively transfer to countersurfaces and wears at a high rate, but the wear can be reduced by using a lubricous coating such as dry lubricant film. In relation to this finding, Dong et.al point out that titanium alloy Ti6Al4V shows strong adhesion wear which owing to the electron structure, crystal structure, and high E/H ratio of the titanium alloy (Dong et al., 1997). Furthermore, it was suggested that thermal oxidation treatments can improve the friction and wear of titanium alloy Ti 6Al4V titanium (Dong & Bell, 2000).

The titanium alloy Ti 10V2Fe3Al is a near beta (β) alloy and is known for its notable properties, including heat treatability, cold formability, great manufacturability, superb tensile strength, and good toughness, among others. The aforementioned material has been utilized in many aviation components, such as fan discs in large aircraft engines, helicopter rotors, and aircraft landing gear (C. Veiga et al., 2012; Machai & Biermann, 2011; Williams & Boyer, 2020). According to Mehdi et.al (Mehdi et al., 2016), the Ti 10V2Fe3Al alloy exhibit different type wear mechanisms under dry sliding condition, with the specific type of mechanism being dependent on the magnitude of applied stress. Under low loading conditions, oxidative wear is observed to play a prominent role, whereas under high loading conditions, delamination wear is observed to be dominant. Furthermore, it is observed that near- β titanium alloy Ti 10V2Fe3Al has a lower wear rate compared with other near- β titanium alloy Ti-6Al- 4V alloy (Farokhzadeh & Edrisy, 2016).

Applications of carbon nanotubes (CNTs) materials to improve tribological properties of materials have been considered in the past years (Cui et al., 2022; Khanna et al., 2021; Nyholm & Espallargas, 2023; Rahman et al., 2022; Su et al., 2018). The CNTs can be employed as reinforcing particles to increase surface properties (González et al., 2023; Hayashi & Endo, 2011), as lubricant oil additives (Salah et al., 2017), and as solid lubricant (Reinert et al., 2017), The CNT also shows good response on the improvement of tribological properties of grease. Senatore et.al (Senatore et al., 2021) point out that CNTs nanogrease has 30% reduction on frictional coefficient compared to conventional greases. The effectiveness of type of CNT's to improve tribological properties affected by the their medium as well. Cornelio et.al conclude that addition of MWCNT's in oil at a concentration of 0.01% gives the best tribological responses for steel-to-steel contact while the SWCNT's at a concentration of 0.05% in water show best tribological response (Cornelio et al., 2016). Therefore, CNTs material can be considered as a promising material to improve friction and wear properties of titanium alloys. In addition, the study on the lubricated friction and wear

of Ti 10V2Fe3Al is still relatively slow although there is progress in the research on friction and wear of titanium alloy materials in major scope.

Statistical design of experiment (DOE) frequently used to evaluate the significance and relationship of one or more controlled parameters to uncontrolled parameters by arranging combination of the parameters to such a way so their significance and relationship can be concluded. As the number of elements and their values increases, the potential number of tests required becomes impractically vast. Conducting such a large number of studies may lead to heterogeneity or variances, ultimately compromising the accuracy of the experimental results. Fractional replicate designs, also known as fractional factorial designs, have been devised by statisticians in order to effectively tackle the aforementioned concerns (Krishnaiah & Shahabudeen, 2012). Taguchi method is a method that formalized the fractional factorial design of experiment method by orthogonal arrays (OA), which reduced the number of required experiments significantly (Islam & Pramanik, 2016). Taguchi method offers OA designs to have a smaller number of experimental trials leading to savings in resources and time. Therefore, this method has been acknowledged as one of methods that significantly reduced the experimental work while providing reliable results. Taguchi DOE method systematically planned experiment that enables the selection of parameter that exhibits enhanced consistency in its performance within the operational environment. In Taguchi designs, the factors that cannot be controlled and have the potential to influence the outcome of a study are typically referred to as noise factors and Taguchi designs aim to discover controllable parameters that effectively mitigate the impact of noisy factors. Therefore, this work is subjected to study the effect of load, sliding speed and CNT nano-lubricant to friction and wear of Titanium alloy Ti10V2Fe3Al by Taguchi Method and to study wear surface morphology of Titanium Alloy Ti10V2Fe3Al under CNT's nano lubricants.

2.0 EXPERIMENTAL PROCEDURE

2.1 Materials

The Ti-10V-2Fe-3Al alloy sample with hardness of 43.4 ± 1.23 HRC was employed as sample. The sample was prepared by cutting a 12-mm thick Ti 10V2Fe3Al plate into into $\varnothing 4$ mm \times 12 mm pin. The pin sample then slid against AISI 52100 steel disc of 64 ± 0.45 HRC hardness. Prior to the tribological test, both pin and the disc samples were polished with #1200 SiC paper.

Three (3) types of carbon nano tubes materials i.e., Single Wall (SW), Double wall (DW), and multiwall (MW) CNTs were employed in this study. The CNT sample was obtained from Nanjing Co.Ltd, China and used as received. Figure 1 shows the micrograph of the CNTs samples with diameter of 27-50 nm and agglomerated CNT's are observed on all CNT's samples. Additionally, the EDX analysis which shows the respective element contained in the CNT's samples are shown in Table 1.

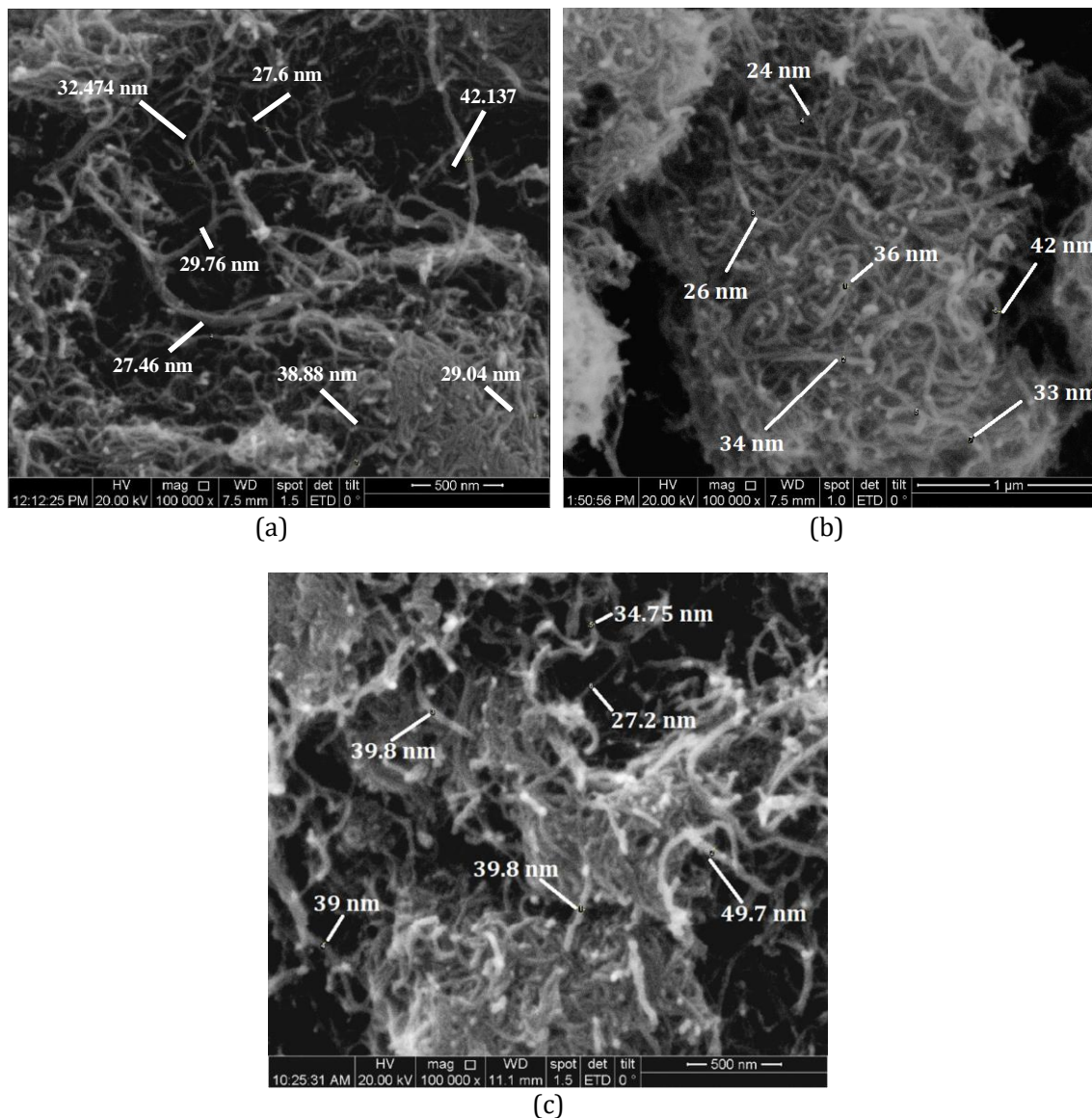


Figure 1: Micrograph of nano particles samples (100k ×mag.); (a) single wall CNTs, (b) double wall CNTs, and (c) multiwall CNTs with their respective tube diameter size.

Table 1: EDX elemental analysis of CNT's samples.

Element	SWCNT		DWCNT		MWCNT	
	Wt%	At%	Wt%	At%	Wt%	At%
CK	87.39	90.5	86.74333	89.71333	90.27333	92.52
OK	12.61	9.77	13.25667	10.29	9.726667	7.48

A commercially available SAE 5w-30 synthetic based oil ($\nu = 73.95 \text{ mm}^2/\text{s} @40^\circ\text{C}$) was employed as lubricant base. The utilization of this fully blended lubricant is based on its comprehensive additives blend content which possibly can contribute to the solubility of the CNT's samples with the oil. The oil was contaminated with 1 ppm CNT of different types of structure, i.e. Single Wall (SW), Double wall (DW), and multiwall (MW) respectively. The nano lubricant samples were prepared by mixing the motor oil with 1 ppm CNT's of different type of structures and stirred with a Magnetic Stirrer at 1250 rpm speed for a duration of 20 minutes. The subsequent step involved subjecting the lubricant blend to sonic agitation at a frequency of 30 kHz using a Sonobio ultrasonic homogenizer for a duration of 30 minutes at ambient temperature (20°C). The mixture was then left undisturbed for several nights to ensure that no precipitation of carbon nanotubes (CNTs) occurred in the lubricant blend. The nano lubricant samples that have been developed are depicted in Figure 2. To simplify the notation, the nano lubricant blend is named Lube 1 (SAE 5w-30/SWCNT), Lube 2 (SAE 5w-30/DWCNT) and Lube 3 (SAE 5w-30/MWCNT) respectively. Moreover, the dynamic viscosity of the samples measured by NDJ-8S rotational viscometer at ambient temperature is shown in Figure 3.

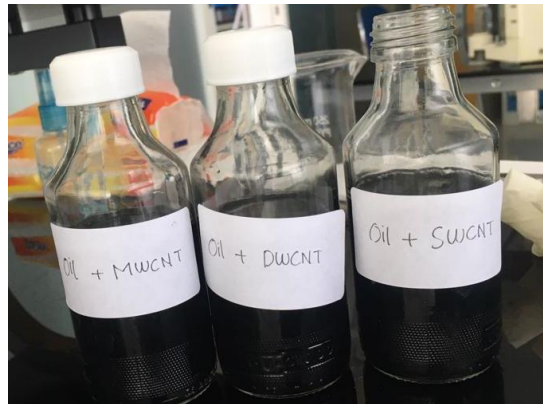


Figure 2: Nano lubricant samples.

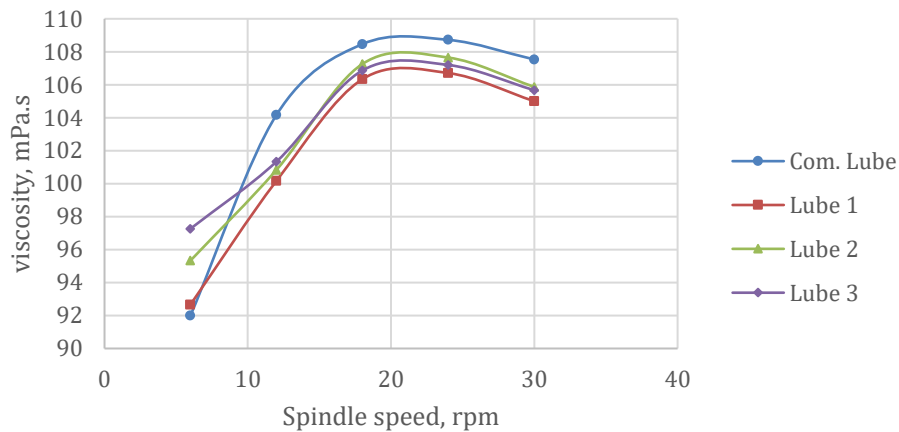


Figure 3. Dynamic Viscosity of Lube sample at various spindle speeds.

2.2 Tribological Properties Evaluation

Friction and wear of the sample was evaluated by GUNT TM-260.03 pin-on-disk apparatus in accordance with ASTM G99 - Standard Test Method for Wear Testing with a Pin-on-Disk Apparatus. The schematic view and real test set up of the apparatus is shown in Figure 4. In present work, a stationary pin was slid against a rotating disc at wear track diameter of 3-cm thus given average periphery of wear track of 0.0942-m. Both pin and disc were polished with #1200 grid SiC paper prior to each testing run to give a similar surface condition and cleaned with acetone after the test.

Friction force was acquired by a load cell attached to the loading arm of the apparatus and displayed on a display with respect to testing time. Therefore, the friction coefficient (μ) can be calculated from the ratio of friction force (F) to normal load (N) applied to the sample through the loading arm by formula:

$$\mu = \frac{F}{N} \quad (1)$$

The wear loss is calculated as the differences in wear pin height before and after the test which is measured by a Mitutoyo digital caliper with 0.01 mm resolution. The wear rate was determined from the wear loss over the sliding distance. Replication of testing was performed based on Taguchi's design of experiment matrix.

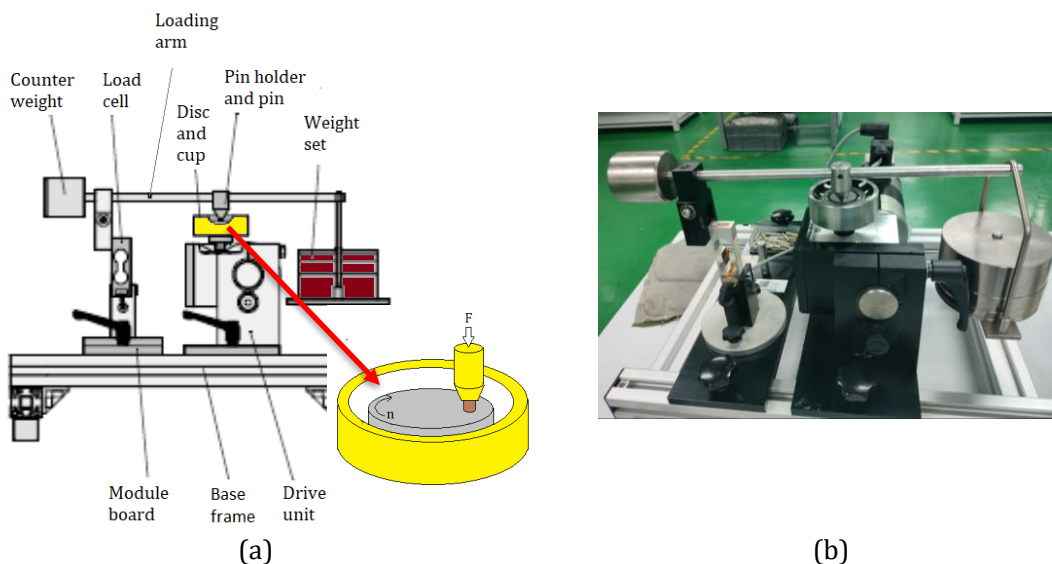


Figure 4: Schematic view test set up (a) and real set up (b) of the pin on disk test.

2.3 Taguchi Design Matrix

In this work, the Taguchi method was employed to determine which control parameter may reduce the friction and wear rate without increasing the variability on the friction and wear rate. Taguchi L9 orthogonal array design consists of three factors at three level was employed. The Taguchi and variance analysis were performed by using Minitab software Three (3) testing

parameters were employed as controlled parameter i.e.: (i) The CNT (nano-oil) type, i.e. Lube 1, Lube 2, and Lube 3, (ii) The normal load i.e. 40N, 60N and 80 N, and (iii) Disc rotational speed i.e. 120 rpm, 150 rpm and 180 rpm. The interaction of these parameters to two uncontrolled parameters, i.e. friction and wear, was investigated. Each experiment was conducted twice, with a distinct experimental order each time. Table 1 displays the L9 design matrix of the experiment, which includes the variables and their corresponding levels.

The S/N ratio is typically employed as a quantitative analytical technique in Taguchi analysis to optimize the output. Taguchi categorizes quality characteristics into three distinct groups: , (i) the smaller the better, (ii) the larger the better, and (iii) the nominal the better. In the present study, the principle of "smaller S/N ratio the better" is employed to evaluate both friction and wear characteristics. The determination of the attribute of smaller being better is based on formula:

$$SN = -10 \log \left(\frac{1}{n} \sum_{i=1}^n Y_i^2 \right) \tag{2}$$

Where Y is the responses for the given factor level combination and n is the number of responses in the factor level combination.

Table 1: Taguchi’s L9 orthogonal matrix of variable and levels.

Experiment No	Lube	Load	Speed	Friction coefficient		Wear rate (mm/m)	
	Type	(N)	(rpm)	Run 1	Run 2	Run 1	Run 2
1	1	20	120				
2	1	30	150				
3	1	40	180				
4	2	20	150				
5	2	30	180				
6	2	40	120				
7	3	20	180				
8	3	30	120				
9	3	40	150				

2.4 Morphological Analysis

FEI Inspect-S50 Scanning Electron Microscope (SEM) with Energy dispersive X-Ray (SEM-EDX) was employed to study the CNT features and their elements as shown in Figure 1 while Thermo Scientific Phenom-ProX Scanning electron microscope (SEM) was employed to study wear surface of the pin samples. In addition, energy dispersive X-Ray (EDX) analysis was performed to evaluate the elements built on the rubbing surface of pin samples.

3.0 RESULTS AND DISCUSSION

3.1 Taguchi Analysis

The Taguchi method typically applied as a tool for optimizing the number of variables where only a few variables contribute significantly, and their interaction effects are relatively low. Taguchi method uses Signal-Noise (S/N) ratio as the quality characteristic of choice where the S/N ratio represent the desirable signal value and the undesirable signal value (Shahavi et al., 2016). In present work, the smaller the better categories were employed to observe interaction of the control factor i.e. Lube type (CNT's type), applied load, and disc rotational speed to the uncontrolled factor (noise) i.e. wear and friction. . Table 2 shows the Taguchi's matrix of variables and their effects on coefficient of friction and wear where we can observe the variability of friction and wear to the controlled parameters.

Table 2: Taguchi's matrix of variable and levels affects the coefficient of friction (CoF) and wear.

Lube Type	Load (N)	Speed (rpm)	Friction coefficient		Wear rate (mm/m)	
			Run 1	Run 2	Run 1	Run 2
1	20	120	0.290	0.210	0.000030	0.00118
1	30	150	0.267	0.343	0.000142	0.003444
1	40	180	0.963	0.730	0.005426	0.002516
2	20	150	0.275	0.265	0.000048	0.004907
2	30	180	0.980	0.740	0.003381	0.002398
2	40	120	1.003	0.568	0.004659	0.00059
3	20	180	0.255	0.250	0.000034	0.000197
3	30	120	1.001	0.827	0.003067	0.003539
3	40	150	0.905	0.720	0.006134	0.004718

Table 3 shows the Signal to Noise (S/N) Ratios response for friction coefficient. It can be observed that the applied load with delta of 10.121 is ranked as factor has the largest effect on the S/N ratio of friction, this interaction significance then followed by lube type (CNT's type) with delta 3.056 and sliding speed (delta =2.971).. Figure 5 (a) shows the main effect plot for S/N Ratio to coefficient of friction where we can observe significant signal to noise ratio differences on the load to friction. The load of 20 N shows the highest S/N ratio thus giving smaller friction variability between the control parameters. Experiments run with Lube 1 show higher S/N ratio for friction compared to other lubes, thus the use of Lube 1 shall give smaller variability to friction coefficient.

Table 4 shows the Signal to Noise (S/N) Ratios response for wear rate where we can observe the load is ranked as factor has the significant effect on the S/N ratio for wear rate with Delta value of 15.15. The following factor is the speed with delta value of 9.45 and lube type (CNT's type) with delta 7.46. Figure 5 (b) shows the main effects plot for S/N ratio for wear rate, it can be observed the experiment runs with load of 20 N shows higher S/N ratio for wear rate thus it will give smaller variability for wear. In terms of lube types, the experiment runs with the Lube 3 shows a higher S/N ratio compared to Lube 1 and Lube 2 for wear. Therefore, the use of this Lube will provide smaller variability to wear rate. Furthermore, the experiment runs at 180 rpm speed,

which is equivalent with 508.7 mm/s linear speed, shows a higher S/N ratio for the wear rate, thus experiment with this speed will give smaller variability to wear rate.

Table 3: Response Table for S/N Ratios for friction.

Level	Lube type	Load (N)	Speed(rpm)
1	7.849	11.752	4.818
2	4.792	4.071	7.788
3	4.812	1.632	4.850
Delta	3.056	10.120	2.971
Rank	2	1	3

Table 4: Response Table for Signal to Noise Ratios for Wear rate.

Level	Lube Type	Load (N)	Speed (rpm)
1	53.77	62.58	53.58
2	49.81	50.84	48.90
3	57.27	47.43	58.37
Delta	7.46	15.15	9.47
Rank	3	1	2

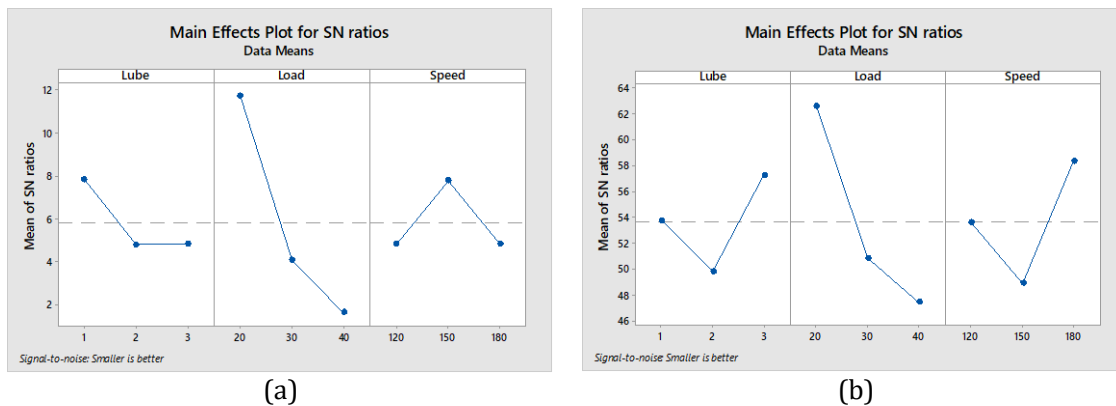


Figure 5: Main effects plot for S/N ratio: (a) S/N ratio for friction, and (b) S/N ratio for wear rate.

Analysis of variance (ANOVA) was performed to determine which factors statistically affect the S/N ratio for coefficient of friction and wear rate by using significance level α ($\alpha = 10\%$). Table 5 and Table 6 show the results of the results of the ANOVA for the friction and wear respectively. The load is the only factor that is considered statistically significant to the S/N ratio for friction of Titanium Ti10V2Fe3Al with the p-value of 0.114. However, all control factors are considered not statistically significant to S/N ratio for wear rate of the titanium sample with p-value more than 0.1.

Table 5: Analysis of Variance for S/N ratio to coefficient of friction.

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Lube Type	2	18.55	9.273	0.86	0.538
Load	2	167.37	83.685	7.74	0.114
Speed	2	17.46	8.729	0.81	0.655

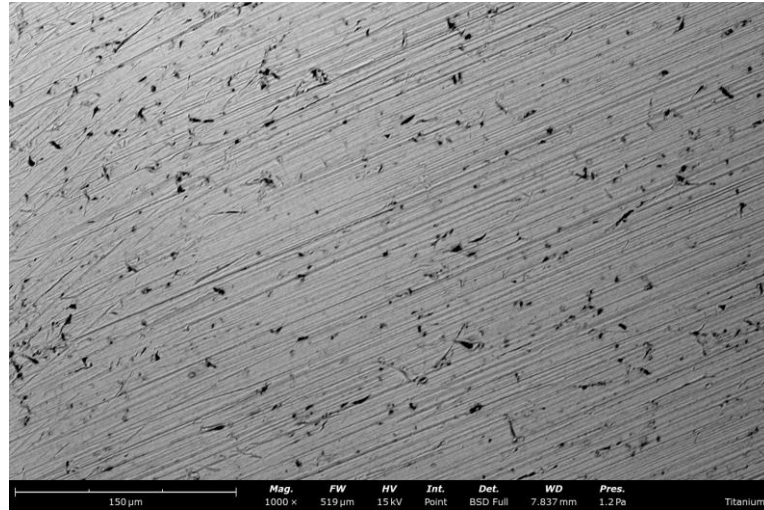
Table 6: One way ANOVA analysis of the test variables to wear rate.

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Lube Type	2	83.58	41.79	0.46	0.685
Load (N)	2	378.94	189.47	2.08	0.325
Speed (rpm)	2	134.53	67.26	0.74	0.575

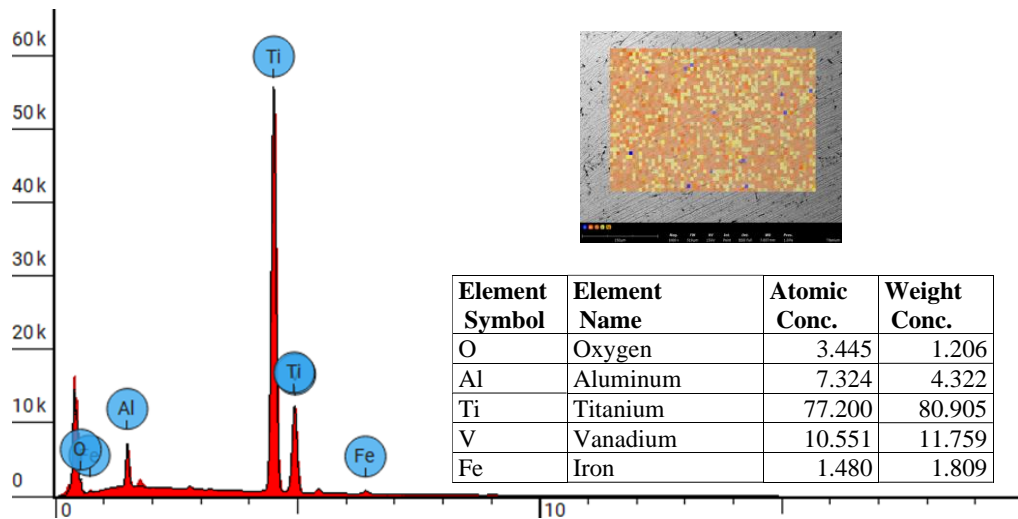
3.3 Morphology and Elemental Analysis

Figure 6 shows the SEM micrograph and EDX examination of the titanium pin initial surface. Ti 10V-2Fe-3Al alloy typically consist of 9.25 – 10.75 % of Vanadium, 1.6 – 2.5% of Iron, 3.75 – 5.25 % Aluminum, as main alloying components (Lampman, 1990) and the presence of these elements also observed in EDX analysis as shown in Figure 6 (b).

The wear surface morphology typically studied to understand wear processes that have been taken place during the sliding. Figure 7 shows the worn surface morphology of the titanium alloy pin lubricated with Lube 1 under the highest testing load and Figure 8 shows the EDX observation at three different points on the worn pin sample lubricated with Lube 1. Strong adhesive wear indication can be observed on the titanium pin surface. This feature can be observed from the flake like wear debris attached on the top surface of pin which represent there was adhesion wear processes have been taken place. Trace of carbonaceous component which was not available on the initial surface morphology was also observed. This carbonaceous component represented by local darker spots on the worn surface and high amount of carbon is also confirmed by the EDX analysis performed on these spots as shown in Figure 8. Figure 8(a) shows the EDX analysis at a point on pin surface, where the primary element of Ti-10-2-3 such as Ti, V, and Al are observed. However, a relatively high percentage of carbon content was detected in this location while no other elements from typical lubricant additives such as Mg, Zn, Ca, Si, P, etc. are observed. High amount of carbon element also detected on the other location of the surface as shown in Figure 8(b), which combined with Fe and small percentage of O and Ti. High percentage of carbon observed on this location suggesting that the carbon from the nano-lubricant has been absorbed to the titanium surface to provide additional surface protection from friction and wear. Moreover, Figure 8 (c) shows the EDX analysis on the adhered wear debris where high percentage of Ti and C were detected, accompanied by the other original element of the titanium sample such Al, Fe, O, and V. This debris suggests that the pin experience significant surface adhesion wear where debris which originated from other location attached to this location.



(a)



(b)

Figure 6: Micrograph of morphology titanium alloy pin sample before testing (a) and its respective EDX analysis showing elements contained at the pin sample (b).

Several mechanisms have been proposed by researchers on the tribological properties enhancement mechanism of nanoparticles as lubricant additives. Ghaednia and Jackson (Ghaednia & Jackson, 2013) proposed that the nanoparticles will separate the contacting surfaces, resulting in void areas in the vicinity of particles and each nanoparticle in contact areas will act as a nano-indenter and will be able to deform the surfaces. Under high contact pressure, the CNT's can deform and shift to lamellar shape to act form transfer layer on the surface of sliding pair (Cornelio et al., 2016; Ni & Sinnott, 2001). CNT's typically has tubular structure so it possibly act as a roller bearing transform the sliding into rolling which minimizing the shear stress on the contact asperities, bear the external load between mating surfaces like cushions, fix the surface

defects to lower stress concentrations and slowing down the propagation of cracks, reduce the surface roughness as polishing agents and form tribo-films with low shear resistance (Ye et al., 2019); (Wang et al., 2022). In addition, high Young's modulus and tensile strength possessed by the CNT's due to their honeycomb like carbon arrangement is considered to give additional strength for load bearing thus reducing the wear.

In relation to the above phenomenon, schematic illustration of typical surface is presented in Figure 9. Since there is no totally smooth solid surface, presence of surface "peak and valley" will always exist in every surface of solid. The surface asperities make micro or nano interlocking between surface are possible to be happened, thus the nano particles are possible to rolled between asperities to change sliding action between tip of the mating surfaces to be rolling action. Therefore, the locally rough surface feature shown in Figure 6 (a) possibly has the role to the CNT's interaction to pin surface during the sliding friction as well. Hence, we believe that the CNT's assisting the lubricant to separate the contact asperities during the sliding friction by rolling action. Since the SWCNT's commonly possess a single tubular layer of carbon, the nanoparticles possibly exfoliated then attached to the pairing surface during the frictional contact to give smaller variability to friction. However, there is also possibility that the CNT's bundles entangled along the contact surface to make it difficult to roll, exfoliate, and dispersed (Nyholm & Espallargas, 2023; Tasis et al., 2006). By observing the CNTs micrograph in Figure 1, this postulation can be a reason for the lower S/N ratio for friction in Lube 2 and Lube 3. However, similar reasons also explain why the Lube 3 has high S/N ratio for wear to provide cushioning effect to the surface.

Figure 10 shows the worn pin sample lubricated with Lube 2 at the load of 40N and 180 rpm speed. Adhesion wear debris incorporated with several carbon spots are also observed on this pin surface. Figure 11 shows the EDX observation at three different points on the worn pin sample lubricated with Lube 2 at the load of 40N and 180 rpm speed. Figure 11(a) shows the EDX analysis at a point on the worn surface where elements of Ti, C, Al, and V are observed on this location. It also can be observed that a high concentration of carbon exists on this location which indicates the absorption of carbon into the Ti pin surface. EDX analysis on wear debris in Figure 11(b) shows significant increasing amount of Fe and O suggesting that surface components from the steel disc have been adhered to the pin due to adhesion process to form surface oxides at this location. Furthermore, we can observe the presence of boron, nitrogen, and sodium accompanying high amount of carbon in Figure 11 (c), which possibly originated from the additive's packages contained in the lubricant base.

The worn surface of Ti alloy pin lubricated with Lube 3 is shown in Figure 12 which shows that the adhesive wear still a dominant mechanism to wear out of the titanium pin. . Although Taguchi analysis suggesting that the lube 3 shows smaller variation to wear rate, a large wear fragment showing different debris layer attached on another layer of debris can be observed in Figure 13 (a) and (b) suggesting strong adhesion mechanism. Figure 13 shows the EDX analysis performed on the wear debris which identifies the presence of elements of C, O, Al, Ti, V, and Fe similar to elements found in other types of nano lube samples. Figure 13(a) and Figure 13 (b) shows the EDX analysis of the inner layer and outer layer of the debris where high content of C and Ti accompanied with O, Al, and V were detected. Additionally, Figure 13 (c) shows EDX analysis from other location from pin surface suggesting high concentrations of C and a notable amount of O also presence suggesting surface oxides have been formed at this location.

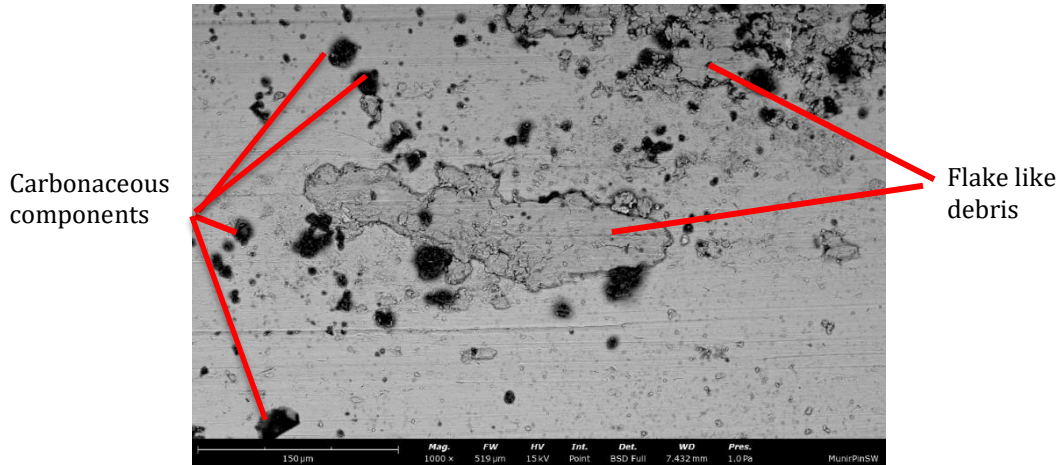
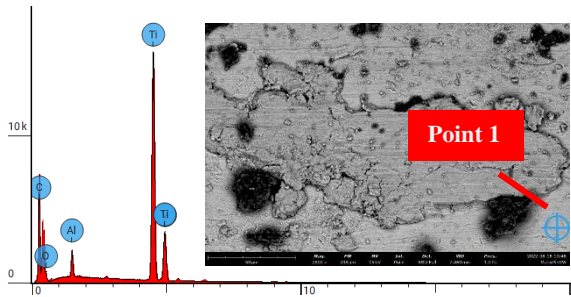
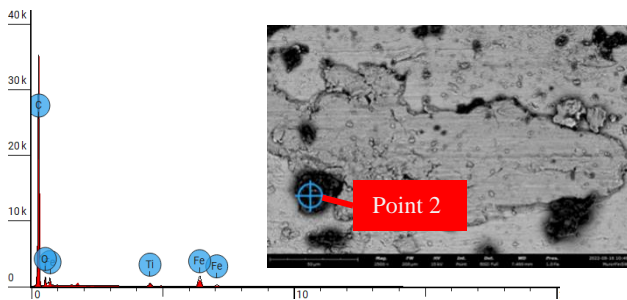


Figure 7: Morphology of worn titanium alloy pin samples lubricated with Lube 1 at the load of 40N and 180 rpm speed.



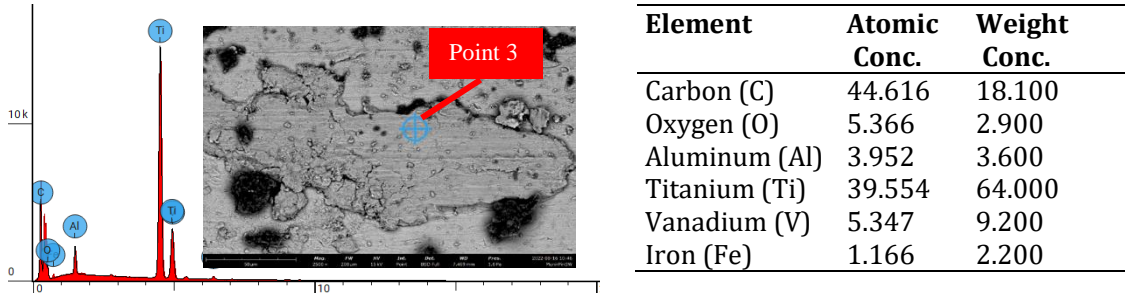
Element	Atomic Conc.	Weight Conc.
Carbon (C)	51.056	21.179
Aluminum (Al)	3.754	3.497
Titanium (Ti)	39.853	65.934
Vanadium (V)	5.337	9.391

(a) Point 1



Element	Atomic Conc.	Weight Conc.
Carbon (C)	92.553	75.956
Oxygen (O)	1.472	1.610
Titanium (Ti)	0.676	2.213
Iron (Fe)	5.299	20.221

(b) Point 2



(c) Point 3

Figure 8: EDX spectra at three different points on the worn pin sample lubricated with Lube 1 at the load of 40N and 180 rpm speed.

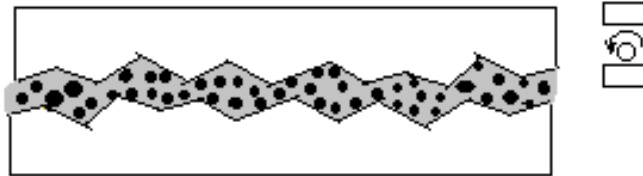


Figure 9: Schematic illustration of CNTs rolling during contact sliding.

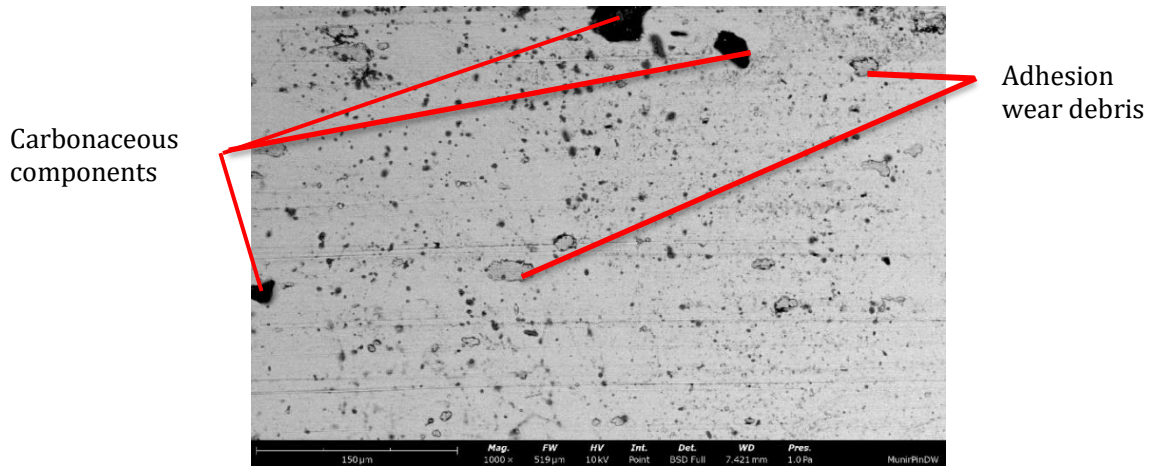
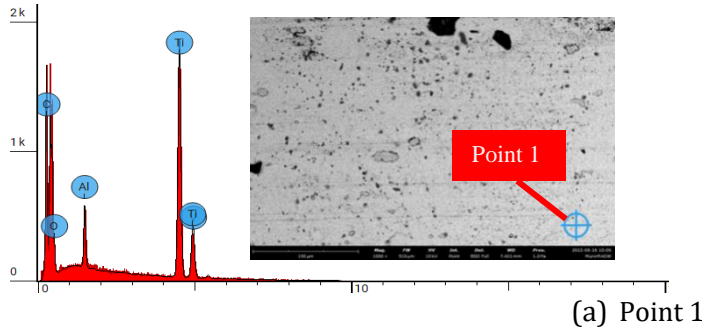
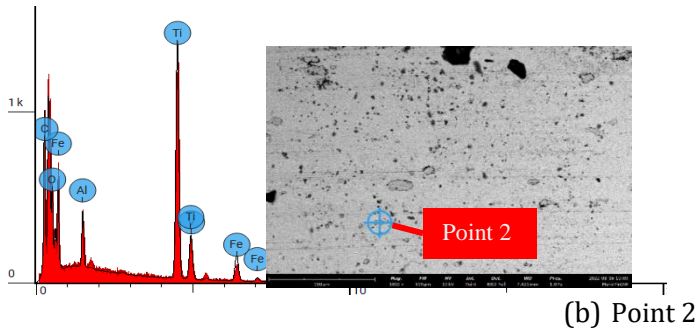


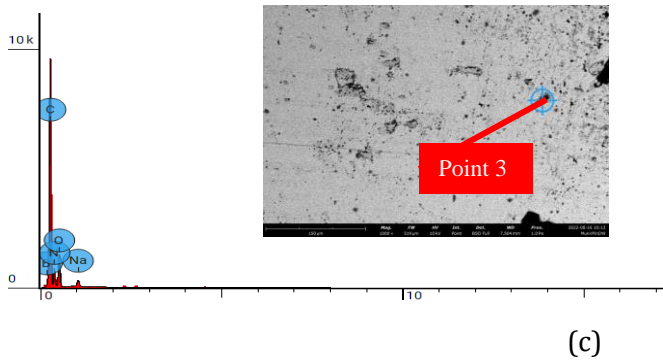
Figure 10: Morphology of worn titanium alloy pin samples lubricated with Lube 2 at the load of 40N and 180 rpm speed.



Element	Atomic Conc.	Weight Conc.
Carbon (C)	32.893	11.189
Aluminum (Al)	5.100	3.896
Titanium (Ti)	52.797	71.628
Vanadium (V)	9.209	13.287



Element	Atomic Conc.	Weight Conc.
Carbon (C)	21.668	6.593
Oxygen (O)	5.175	2.098
Aluminum (Al)	3.947	2.697
Titanium (Ti)	43.463	52.747
Vanadium (V)	4.567	5.894
Iron (Fe)	21.180	29.970



Element Name	Atomic Conc.	Weight Conc.
Boron (B)	3.779	3.103
Carbon (C)	53.658	48.949
Nitrogen (N)	28.220	30.030
Oxygen (O)	13.426	16.316
Sodium (K)	0.917	1.602

Figure 11: EDX spectra at two different locations on the worn pin sample lubricated with Lube 2 at the load of 40N and 180 rpm speed.

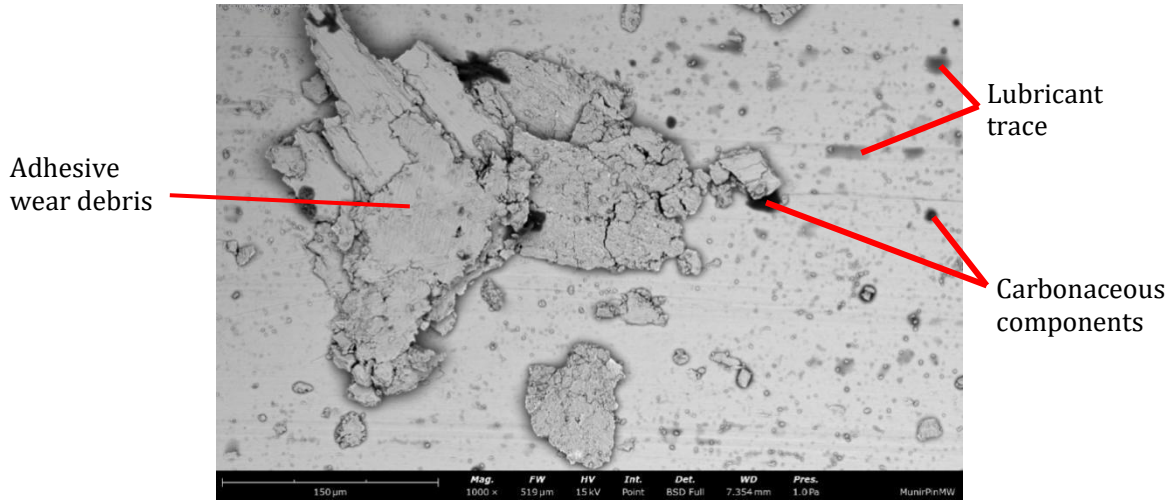
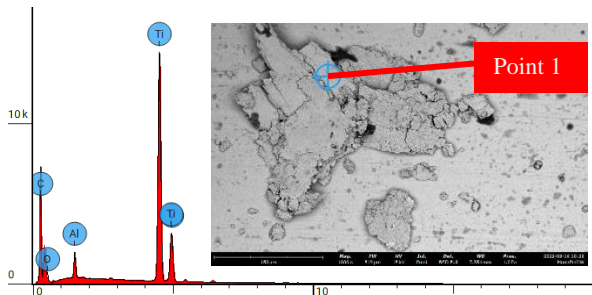
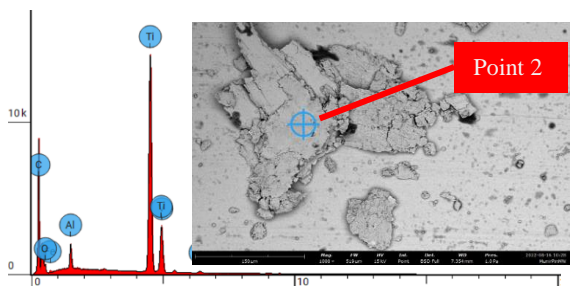


Figure 12: Morphology of worn titanium alloy pin samples lubricated Lube 3 at the load of 40N and 180 rpm speed.



(a) Point 1

Element Name	Atomic Conc.	Weight Conc.
Carbon	53.583	22.900
Aluminum	3.439	3.300
Titanium	37.958	64.700
Vanadium	5.020	9.100



(b) Point 2

Element Name	Atomic Conc.	Weight Conc.
Carbon	57.714	25.926
Aluminum	3.076	3.103
Titanium	34.026	60.961
Vanadium	4.466	8.509
Iron	0.719	1.502

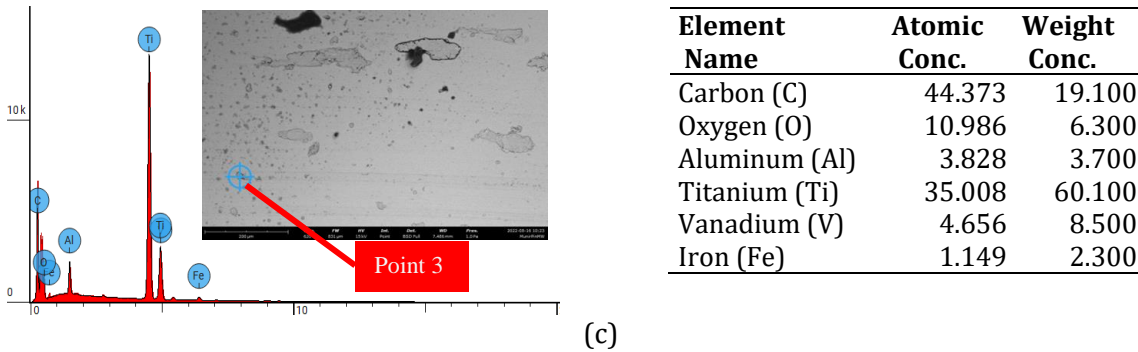


Figure 13. EDX spectra at two different locations on the worn pin sample lubricated with Lube 3 at load of the 40N and 180 rpm speed.

SEM micrograph observation on the titanium alloy samples suggesting that the lubricated wear of the titanium alloy sample dominantly caused by the adhesion which similar to most titanium material. The adhesion mechanism itself is a very important process in friction and wear process. The friction coefficient typically depends on the flow of stress on the surface asperities where the deformation to accommodate rubbing takes place, consequently, the surface asperities break down and wear taken place. As the process of sliding continued, the surface asperities frequently crack and attached to other sites of surface asperities to form adhesion layer. Titanium has a very high degree of unsaturation bond, thus shows a strong tendency to bond with almost anything, such as a matching titanium surface or a nonmetal such as lubricant. As the degree of d-bond character increases, the friction coefficient decreases because the greater the degree of bonding of a metal to itself, the less the bonding across the interface (Buckley, 1981; Larsen-Basse, 1992). The interaction of CNT's and operating parameter such as load and speed to titanium surface may initiate the rolling action of CNT'S, thus exfoliate the CNT structure and introduce the absorption of CNT's carbon layer to titanium surface to form a protective mechanism to reduce the friction and wear.

CONCLUSIONS

Based on the Taguchi Method analysis, we can conclude that the operating load is the significant factor that gives low variability to friction and wear of Ti 10V2Fe3Al lubricated with CNT's nano lubricant compared to speed and CNT's type. A lower load gives less friction and wear variability compared to a higher load. The use of different types of CNT's may give different effects to friction and wear of titanium alloy. The SWCNT's (Lube 1) shall provide smaller variability to friction but the use of MWCNT (Lube 3) gives smaller variability to wear rate. Moreover, speed also gives different effect to variability of friction and wear of titanium alloy. Experiment runs at 150 rpm rotational speed, which in this case equivalent with 235.5 mm/s of linear speed, shows smaller variability to friction coefficient while experiment runs with 180 rpm speed (equivalent with 508.7 mm/s linear speed) give smaller variability to wear rate. The interaction of CNT's and operating parameter such as load and speed to titanium surface may initiate the rolling action of CNT'S, thus exfoliate the CNT structure and introduce the absorption of CNT's carbon layer to titanium surface to form a protective mechanism to reduce the friction and wear. Therefore, the use of various types of CNT's blended with commercial motor oil may generally act as friction modifier and bear the external load between mating surfaces to reduce the friction and wear.

However, surface micrograph observation from pin sample that experiencing high load suggesting that the adhesive wear process may still dominantly taken place although nano lubricant still presence during the lubricated friction of Ti 10V2Fe3Al.

ACKNOWLEDGMENTS

The authors would like to thank Universitas Negeri Malang for funding this work under Hibah Kerjasama Internasional 2022 No. 19.5.1254/UN32.20.1/LT/2022 and Universitas Muhammadiyah Surakarta for supporting this work through Hibah Integrasi Tridharma PT No 233 /A.3-III/FT/VI/2022.

REFERENCES

- Buckley, D. H. (1981). Surface Effects in Adhesion, Friction, Wear, and Lubrication. In ASM Handbook Vol. 18.
- Budinski, K. G. (1991). Tribological properties of titanium alloys. *Wear*, 151, 203–217.
- C. Veiga, J.P. Davim, & A.J.R. Loureiro. (2012). Properties and Applications of Titanium Alloys: a Brief Review. *Reviews on Advanced Materials Science*, 32(2), 133–148.
- Cornelio, J. A. C., Cuervo, P. A., Hoyos-Palacio, L. M., Lara-Romero, J., & Toro, A. (2016). Tribological properties of carbon nanotubes as lubricant additive in oil and water for a wheel–rail system. *Journal of Materials Research and Technology*, 5(1), 68–76. <https://doi.org/10.1016/j.jmrt.2015.10.006>
- Cui, X., Li, C., Ding, W., Chen, Y., Mao, C., Xu, X., Liu, B., Wang, D., Li, H. N., Zhang, Y., Said, Z., Debnath, S., Jamil, M., Ali, H. M., & SHARMA, S. (2022). Minimum quantity lubrication machining of aeronautical materials using carbon group nanolubricant: From mechanisms to application. *Chinese Journal of Aeronautics*, 35(11), 85–112.
- Dong, H. (2010). Tribological properties of titanium-based alloys. In H. Dong (Ed.), *Surface Engineering of Light Alloys: Aluminium, Magnesium and Titanium Alloys* (pp. 50–58). Woodhead Publishing, UK.
- Dong, H., & Bell, T. (2000). Enhanced wear resistance of titanium surfaces by a new thermal oxidation treatment. *Wear*, 238, 131–137.
- Dong, H., Bloyce, A., Morton, P. H., & Bell, T. (1997). Surface Engineering To Improve Tribological Performance Of Ti-6Al-4V. *Surface Engineering*, 13(5), 402–406.
- F.H. Froes. (2015). *Titanium—Physical Metallurgy, Processing, and Applications* (F. H. Froes, Ed.). ASM International.
- Farokhzadeh, K., & Edrissy, A. (2016). Transition between mild and severe wear in titanium alloys. *Tribology International*, 94, 98–111.
- Ghaednia, H., & Jackson, R. L. (2013). The Effect of Nanoparticles on the Real Area of Contact, Friction, and Wear. *Journal of Tribology*, 135(4). <https://doi.org/10.1115/1.4024297>
- González, J., Ghaffarinejad, A., Ivanov, M., Ferreira, P., Vilarinho, P. M., Borrás, A., Amorín, H., & Wicklein, B. (2023). Advanced Cellulose–Nanocarbon Composite Films for High-Performance Triboelectric and Piezoelectric Nanogenerators. *Nanomaterials*, 13(7), 1206. <https://doi.org/10.3390/nano13071206>
- Hayashi, T., & Endo, M. (2011). Carbon nanotubes as structural material and their application in composites. *Composites Part B: Engineering*, 42(8), 2151–2157. <https://doi.org/10.1016/j.compositesb.2011.05.011>

- Kaur, S., Ghadirinejad, K., & Oskouei, R. H. (2019). An Overview on the Tribological Performance of Titanium Alloys with Surface Modifications for Biomedical Applications. *Lubricants*, 7(65). <https://doi.org/doi:10.3390/lubricants7080065>
- Khanna, V., Kumar, V., & Bansal, S. A. (2021). Effect of carbonaceous nanomaterials' reinforcement on mechanical properties of aluminium metal-based nanocomposite: A review. *Materials Today: Proceedings*, 38(1), 289–295. <https://doi.org/10.1016/j.matpr.2020.07.221>
- Krishnaiah, K., & Shahabudeen, P. (2012). *Applied Design of Experiments and Taguchi Methods* (1st ed.). PHI Learning Pvt.Ltd.
- Lampman, S. (1990). Wrought Titanium and Titanium Alloys. In *Properties and Selection: Nonferrous Alloys and Special-Purpose Materials* (pp. 592–633). ASM International. <https://doi.org/10.31399/asm.hb.v02.a0001081>
- Larsen-Basse, J. (1992). Basic Theory of Solid Friction. In *ASM Handbook, Volume 18: Friction, Lubrication, and Wear Technology*.
- Machai, C., & Biermann, D. (2011). Machining of a hollow shaft made of β -titanium Ti-10V-2Fe-3Al. 2011 IEEE International Symposium on Assembly and Manufacturing (ISAM), 1–6. <https://doi.org/10.1109/ISAM.2011.5942364>
- Mehdi, M., Farokhzadeh, K., & Edrissy, A. (2016). Dry Sliding Wear Behaviour of Superelastic Ti-10V-2Fe-3Al Titanium Alloy. *Wear*, 350–351, 10–20.
- Nyholm, N., & Espallargas, N. (2023). Functionalized carbon nanostructures as lubricant additives – A review. *Carbon*, 201, 1200–1228. <https://doi.org/10.1016/j.carbon.2022.10.035>
- R.R. Boyer, & Briggs, R. D. (2013). The Use of β Titanium Alloys in the Aerospace Industry. *Journal of Materials Engineering and Performance*, 22(10), 2916–2920.
- Rahman, M. M., Islam, M., Roy, R., Younis, H., AlNahyan, M., & Younes, H. (2022). Carbon Nanomaterial-Based Lubricants: Review of Recent Developments. In *Lubricants* (Vol. 10, Issue 11). MDPI. <https://doi.org/10.3390/lubricants10110281>
- Reinert, L., Lasserre, F., Gachot, C., Grützmacher, P., MacLucas, T., Souza, N., Mücklich, F., & Suarez, S. (2017). Long-lasting solid lubrication by CNT-coated patterned surfaces. *Scientific Reports*, 7(1), 42873. <https://doi.org/10.1038/srep42873>
- Salah, N., Alshahrie, A., Abdel-wahab, M. Sh., Alharbi, N. D., & Khan, Z. H. (2017). Carbon nanotubes of oil fly ash integrated with ultrathin CuO nanosheets as effective lubricant additives. *Diamond and Related Materials*, 78, 97–104. <https://doi.org/10.1016/j.diamond.2017.08.010>
- Shahavi, M. H., Hosseini, M., Jahanshahi, M., Meyer, R. L., & Darzi, G. N. (2016). Clove oil nanoemulsion as an effective antibacterial agent: Taguchi optimization method. *Desalination and Water Treatment*, 57(39), 18379–18390. <https://doi.org/10.1080/19443994.2015.1092893>
- Su, Y., Tang, Z., Wang, G., & Wan, R. (2018). Influence of CNT on Tribological Properties of Vegetable oil. *Research Article Advances in Mechanical Engineering*, 10(5), 2018. <https://doi.org/10.1177/1687814018778188>
- Tasis, D., Tagmatarchis, N., Bianco, A., & Prato, M. (2006). Chemistry of Carbon Nanotubes. *Chemical Reviews*, 106(3), 1105–1136. <https://doi.org/10.1021/cr050569o>
- Wang, B., Qiu, F., Barber, G. C., Zou, Q., Wang, J., Guo, S., Yuan, Y., & Jiang, Q. (2022). Role of nano-sized materials as lubricant additives in friction and wear reduction: A review. *Wear*, 490–491, 204206. <https://doi.org/10.1016/J.WEAR.2021.204206>
- Williams, J. C., & Boyer, R. R. (2020). Opportunities and Issues in the Application of Titanium Alloys for Aerospace Components. *Metals*, 10(6), 705. <https://doi.org/10.3390/met10060705>