TRIBOLOGICAL PERFORMANCE OF GREASE WITH DIFFERENT

TYPES OF ADDITIVES



UNIVERSITI TEKNIKAL MALAYSIA MELAKA

TRIBOLOGICAL PERFORMANCE OF GREASES WITH DIFFERENT TYPE OF ADDITIVES.

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ALAYSIA A report submitted in fulfilments of the requirements for the degree of **Bachelor of Mechanical Engineering with Honours** UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

DECLARATION

I Nur Aqila Syamimi Binti Amir Hamzah declares this project report entitled "Tribological Performance of Grease with Different Type of Additives" under the guidance of Dr. Mohd Rody Bin Mohamad Zin, is my original work except references material.



APPROVAL

I here with declare that I have read this project report and in my opinion this report is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering.



DEDICATION

I dedicated my study to my beloved parent who always gives motivation and encouragement to finish my study. Not to be forgotten to all my siblings who always cheer me up and encourage me to work hard in completing this study. Apart from that, to all my peers who always be with me through thick and thin. Finally, I would like to express my sincere gratitude to my supervisor Dr. Rody Bin Mohamad Zin for his time, guidance, and patience to finish the work and those who always help me to the ends.



ABSTRACT

The tribological properties and lubrication mechanisms of oil need extensive performance on a textured surface. Despite this, the lubrication of grease on textures surfaces is considered as new. This report will present an experimental study of grease in frictional behaviors with lubricated sliding contact under mixed conditions. The experiment is conducted by using a 4-ball tester machine with ASTM D2266 with influences surface texture parameters on frictional properties. The results give the friction coefficient is mostly reliant on texture parameters and density. The test is conducted with greases formulation of the different additives. The aim is to suppress the best friction properties in all experimental conditions which can accumulate more grease and trap wear debris. The reduction friction is attributable to the formation of a stable grease lubrication film composed of the oil film and the hydrodynamic pressure effect of the surface texture. This property demand to increase the mating gap and reduces the probability of asperity contact. This result will help in understanding the tribological behavior of grease on a textured surface and predict the lubrication conditions of sliding bearings for superior operation in machinery.

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ABSTRAK

Sifat tribologi dan mekanisme pelinciran minyak memerlukan prestasi yang luas pada permukaan bertekstur. Walau bagaimanapun, penggunaan pelinciran minyak pada permukaan tekstur dianggap baru. Laporan ini akan menyajikan kajian eksperimental minyak dalam tingkah laku geseran dengan sentuhan gelongsor yang dilincirkan dalam keadaan bercampur. Eksperimen dijalankan dengan menggunakan mesin penguji 4bola dengan ASTM D2266 dengan mempengaruhi parameter tekstur permukaan pada sifat geseran. Hasil kajian menunjukkan bahawa pekali geseran kebanyakannya bergantung pada parameter dan ketumpatan tekstur yang menghasilkan pekali geseran. Ujian dilakukan dengan minyak dari pelbagai bahan tambahan dari formulasi. Tujuannya adalah untuk menekan sifat geseran terbaik dalam semua keadaan eksperimen yang dapat menyimpan lebih banyak serpihan gris dan perangkap. Geseran pengurangan disebabkan oleh pembentukan filem pelinciran gris stabil yang terdiri daripada filem minyak dan kesan tekanan hidrodinamik tekstur permukaan. Permintaan harta tanah ini untuk meningkatkan jurang kawin dan mengurangkan kebarangkalian hubungan asperiti. Hasil ini akan membantu memahami perilaku tribologi minyak pada permukaan bertekstur dan meramalkan keadaan pelinciran galas gelongsor untuk operasi yang lebih baik dalam mesin.

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

TABLE OF CONTENTS

4 5

DECLARATION APPROVAL DEDICATION		i ii iii
ABSTRACT		iv
ABSTRAK		v
ACKNOWLEDGEMEN	ΝT	vi
TABLE OF CONTENT	S	vii
LIST OF TABLES		ix
LIST OF FIGURES		xi
LIST OF SYMBOLS		xii
LIST OF		•••
ABBREVIATIONS		XIII
CHAPTER MALAYS	IA AL	
1. INTRODUCTIO		1
1.1 Research	Background	1
1.2 Problem S	Statement	3
1.3 - Objective	S	4

Research Scope

General Methodology

1.4

1.5

3.

	4	J.L	la 15:5: " in sid	
2.	LITE	RATUR	E REVIEW	6
	2.1	Introdu	ction	6
	2.2	Tribolo	gical Study NIKAL MALAYSIA MELAKA	6
		2.2.1	Automotive Tribology	7
		2.2.2	Importance of engine tribology	8
		2.2.3	Lubrication Regimes in the Engine	10
	2.3	Lubrica	nt	12
	2.4	Greases		12
	2.5	Additiv	es	13
		2.5.1	Type of Additives	14
	2.6	Friction	Coefficient	20
		2.6.1	The Coefficient of Friction Calculation	22
	2.7	Wear M	echanism	23
		2.7.1	Calculation of Specific Wear Rate	24
	2.8	Surface	Roughness	25

METHODOLOGY273.1Overview273.2Sample Preparation283.3Tribological Test293.4Wear Scar and Surface Roughness Observation38

4.	RES	ULT AN	D DISCUSSION	39
	4.1	Introdu	ction	39
	4.2	Experiment Data		39
		4.2.1	Testing Parameter	39
	4.3	Coeffic	ient of Friction	40
		4.3.1	Coefficient of Friction of Each Specimen	40
		4.3.2	Comparison of COF Over Sliding Time	42
		4.3.3	Comparison of Average COF	43
	4.4	Wear A	Analysis	
		4.4.1	Wear Scar Diameter	44
		4.4.2	Specific Wear Rate 4 Types of Specimen	45
		4.4.3	Surface Morphology of Wear Scar	46
	4.5	Surface	Roughness	47
	4.6	The Re	sult Comparison	48

5. CONCLUSION

CON	ICLUSION	50
5.1	Conclusion	50
5.2	Recommendations for Future Studies	51
RENC	CES	52

REFERENCES APPENDIX A **APPENDIX B**

ģ

56 57

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

LIST OF FIGURES

Figures	Title	Page
1.1	Schematic diagram of the 4-ball tester machine	2
1.2	Flow chart of the project	5
2.1	Main components in an internal combustion engine.	7
2.2	Energy consumption developed in an engine.	8
2.3	Distribution of energy consumption in a light-duty vehicle.	9
2.4	Lubrication regimes for engine components and friction	11
2.5	Grease anatomy	13
2.6	(a) SEM images of MoS2 particles at 500×; (b) SEM images of F- PTFE particles at 100×. (S.Bagi et al., 2015)	20
2.7	Coefficient of friction (b) (c) Wear Scar Diameter	21
2.8	The low magnification secondary electron images	22
2.9	Steel ball scars topography by optical profilometer	23
3.1	Flow chart of the experiment	25
3.2	UNIVERSITI TEKNIKAL MALAYSIA MELAKA Types of greases	26
3.3	The schematic of four ball test.	27
3.4	4-ball Testing Machine	28
3.5	(a) Power Supply (b) PC (c) Electronic Controller	29
3.6	(a) Ball bearing (b) Pot (c) Covers (d) Large component	31
3.7	(a) Allen Key Set (b) Pot (c) Slot (d) Fourth ball bearing place	32
3.8	(a) Ball pot (b) Sensor Wire (c) Load hanged	33
3.9	(a) (b) WINCUCOM (c) Electronic Controller	34
3.10	(a) Ball Bearing (b) Allen Key (c) WINCUCOM	35

3.11	3D non-contact profilometer	36
4.1	Coefficient of friction against sliding time with molybdenum additive	39
4.2	Coefficient of friction against sliding time with lithium complex grease additive.	39
4.3	Coefficient of friction against sliding time with sulfhydryl additive	40
4.4	Coefficient of friction against sliding time with potassium additive	40
4.5	Variation of coefficient of friction with different types of additives	41
4.6	Variation of average CoF with different type of additive	42
4.7	Variation of specific wear rate with different additives	44
4.8	Surface morphology of wear scar for different type of additive	46
4.9	Surface roughness Ra of grease with different additives	47
	UNIVERSITI TEKNIKAL MALAYSIA MELAKA	

LIST OF TABLES

Tables	Title	Page
2.1	Roughness based on profile parameters and COF	26
3.1	Design of experiments	32
3.2	Testing parameter for 4 ball	32
4.1	Testing parameter for 4 ball	39
4.2	Specific wear rate	45
4.3	Result Comparison from Test	48



LIST OF ABBREVIATION

	EP	-	Extreme Pressure
Ι	R & D	-	Research and Development
	WSD	-	Wear-Scar Diameter
	MoS2	-	Molybdenum
	SEM	-	Scanning Electron Microscopy
	LCG	-	Lithium Complex Grease
AT MALA	EHL	-	Elasto Hydrodynamic Lubrication
New York	K	-	Potassium
I II	SH	-	Thiol group
Person In	ZDDP	-	Zinc dialkyldithiophosphate
با ملاك	PTFE	<	Polytetrafluoroethylene
		MI	Molybdenum di sulfide
Ling and Mala	SEM LCG EHL K SH ZDDP PTFE MoS2	- - -	Scanning Electron Microscopy Lithium Complex Grease Elasto Hydrodynamic Lubrication Potassium Thiol group Zinc dialkyldithiophosphate Polytetrafluoroethylene Molybdenum di sulfide

LIST OF SYSMBOLS



r - Distance from the center of the contact surface

on the lower balls to the axis of rotation.

CHAPTER 1

INTRODUCTION

1.1 Research Background

Tribology is known as the science and engineering of interacting surfaces in relative motion and the principles of friction, lubrication, and wear. Lubrication is a constituent of tribology in reducing frictional resistance of surface having relative motion under load. Due to the crease of friction, it has been established that surfaces of the bodies are never perfectly smooth.

The lubricating oils are selected considering the various operations conditions like rising of temperature, normal working temperature, working load, and extreme pressure conditions. The industrial revolution using special-purpose oils and greases to lubricate machine components being operated in the environments. There are various types of lubricants which include oil-based, solid, and semi-solid lubricants. Greases are one of the lubricants that commonly used with respect to the chemical mixture and digitization operates under elevated temperatures, higher loads, and long-life service.

The grease is divided into three board categories which are performance additives, inhibitors & stabilizers, and detergents. The performance additives are dispersed in thickeners which make the grease semi-solid that serve as traps for pockets oil constantly released when interacting surface demands. There are several common types of thickener used which are simple soaps, complex soaps, and non-soap thickeners. Lithium-based simple soaps or Li-complex soaps are commonly used while the Sulphur based additives used to enhance wear performance of greases. The Sulphur contains EP additives react with the metal surface to form a conciliatory tribofilm that protects the underlying metal dorm further wear.

The experiment using the parameter of wear scar diameter and friction coefficient. The principal function of lubricants is to control friction, and wear temperature. The standardized method determines the wear of contact and the pressure that leads to lubrication failure. Therefore, the pressure (EP) and anti-wear (AW) additives are used to improve friction and wear behaviors.

The standard schedule presents the conditions and the procedure of the test and the equipment. In the research, it is compared related to the use of the four-ball machine, using American (ASTM D 2266). Metallic dialkyl dithiocarbamates of zinc, lead, and molybdenum are most used as the additive to provide anti-wear protection and inhibit the oxidation of lubricants. The effect of an additive depends on its chemical nature and concentration in the formulation. The aim of the experiment is to determine the influence of wear and weld of greases with different additives by using the four-ball tester as shown in Fig 1.1. The material of the test balls is 100Cr6 steel with a hardness of 24-62 HRC.



Figure 1.1 Schematic diagram of the 4-ball tester machine

1.2 Problem Statement

The important parameter for the application is Load Carrying Capacity of extreme pressure (EP). The viscosity gives anti-wear benefits to lubricants by the wear scar diameter. The increase of viscosity is varying by the percentage of additives. The study of the antiwear additive has been found through the experimental condition to show the anti-wear properties of lubricants. In the experimental, the four-ball testing machines are used with the same parameters but different types of formulation additives.

This study, the additives are known to increase the performance of greases as well as to reduce the capacity of lubricant consumption. Moreover, the power loss can be control by reducing friction lubricants containing oil soluble. From the research, it is stated that the molybdenum present as the additive to the grease forms a low-friction surface film during the operation of a machine under high loads. The main objective of this report is to observe and search the best additives selected to improve the performance of greases formulation.

The tribology related failures compose in mechanical systems about 30 percent failure. The problem needs to be minimized by the best selection of grease formulation for wear and friction consideration. In general, additive use for the contamination control which prevents contact surface that leads to damage to the metal machine surface.

Therefore, greases with the best formulation will reduced friction, heat, and wear behavior. Moreover, the great formulation of grease helps to maximize the machinery's life. Eventually, it saves money, time, and energy to the production industry for efficient and reliable operation.

1.3 Objective

The objective of this project is:

i. To investigate the tribological performance of grease with different type of additive.

1.4 Research Scope

To achieve the objective of this study, there are important tasks that need to be considered. There are important scopes need to be identified to complete this research. In this study, the MNR oil is used in formulating multipurpose grease. In this study, by using the MNR oil formulating grease which is a heavy-duty grease from the recycling base oil. Moreover, this formulated grease is providing high performance at low cost, environmentally friendly, and safe. In addition, in this research, the additive will be added to give an extra special property in the greases. Each additive gives a different property for example anti-wear, anti-corrosion, antioxidant and extreme pressure, and solid lubricant. In this research, the analysis would be done using the 4-ball testing machine with standard ASTM D2266.

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

1.5 General Methodology

In this section will describe the progress of the project to achieve the objective by using the analysis and data gained. The methodology of the project is summarizing in the flowchart in Figure 1.2 below.



Figure 1.2: Flow chart of the project

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In this chapter, the information related to the study is obtained by the previous research and sources from journals, articles, reports, websites, and books. The focus of this chapter is to act as a guideline from previous knowledge and ideas to run the project. The objective achieves by acquiring the tribological studies which more on the lubricant of the component.

This chapter is organized with the following order which in Section 2.2 will describes more on the tribology studies following the 2.3 Lubricant understanding, 2.4 on greases knowledge, and all parameters of the experiment.

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2.2 Tribological Study

In 1966 the UK Department of Education and Science articulates the concept of tribology which encloses the integrative science and technology of surfaces interact in relative motion correlated subjects and practices. Tribology is the Science & Technology of surface contact between surfaces moving connected. Tribology is influential to reduce the frictional resistance of surface with the relative motion under a certain load. Moreover, the concept includes the hydrodynamic, hydrostatic and Elasto Hydrodynamic Lubrication (EHL) exploiting as a lubricant (Dongare, Pr. Dofr. A. D., 2014)

2.2.1 Automotive tribology

The most important component in the motor vehicle is the reciprocating internal combustion engine as shown in Fig.2.1. It also can be found in any transportation including the ground and sea which are motorcycles, scooters, mopeds, vans, trucks, buses, agricultural vehicles, construction vehicles, trains, boats, and ships. The deficiency of reciprocating internal combustion is it has a low thermal and mechanical efficiencies which cause the energy fuel dissipated as heat loss and friction. The internal combustion engine also acts as a contributor to atmospheric pollution which causes the greenhouse effect via carbon dioxide, hydrocarbon, and particulate NOx. (S.C. Tung et al., 2004)



Fig. 2.1: Main components in an internal combustion engine

2.2.2 Importance of engine tribology

Effective lubrication of all movement component can be achieved by the engine tribology. The adverse impact on the environment can be minimized by reducing friction and wear. The task has a wide range of operations which involves the speed, load, and temperature in the engine. There are several methods to improve the tribological performance of the engines which are by reducing fuel consumption, increasing the engine power output, reducing the oil consumption, reduce the harmful exhaust emissions, Improved durability, reliability, reduced maintenance. Fig. 2.2 shown that only 12% of energy in the fuel available for a drive wheel, 15% dissipated as mechanical due to frictional losses.



Figure 2.2: Energy consumption developed in an engine

From the combustion the energy is distributed in an engine and a powertrain system. Apart from that, the friction loss shown as the major portion which is 48% of energy consumption developed in an engine. The portion of 35% stands for acceleration resistance and 17% for the cruising resistance. For the Fiq.2.3 shows that engine friction loss of bearings, piston rings, and piston skirt friction. Approximately the total friction loss of the valve train, crankshaft, transmission, and gears are 66% while the remaining (34%). The frictional losses arising from the rotating bearing followed by the valve train and the auxiliaries. (M.L. McMillan et al, 2004)



Figure 2.3: Distribution of energy consumption in a light-duty vehicle

2.2.3 Lubrication regimes in the engine

Liquid lubricant is the essential operating tribological parameter in the powertrain system in which lubricant film thickness separates the interacting component surfaces. Precisely, the relative magnitude of the lubricant film compared to the combined surface roughness of the two surfaces. It is called a film thickness ratio, or parameter

$$\lambda = \frac{h}{\left(\sigma_{surface1}^2 + \sigma_{surface2}^2\right)^{1/2}}$$
(2.1)

From the equation *h* represents the film thickness calculated through the application of classical thin film analysis taking the surfaces to be smooth. Where σ is the root mean square (RMS) surface roughness. The centerline average surface roughness values, $(Ra_{surface1} + Ra_{surface2})$ in the denominator in place of the root mean square term. The relationship between the coefficient friction and the oil thickness ratio or Summerfield number (= viscosity × speed/load) as shown in figure 2.4. The top box on the left side is a surface contact and the box on the bottom left is a fluid film that separates the surfaces. The middle part is the extremes partial or intermittent contact occurs.

The curved line below the lubrication regimes expresses the relationship between the friction coefficient and the Summerfield number. During a single cycle, different automotive components depend on different modes of lubrication to achieve acceptable performance. The journal and thrust bearings are designed to operate in the hydrodynamic lubrication regime with lubricant film separate bearing surfaces. Conventional, at the low, seep the actual metal-to-metal contact takes place with high loads and low-viscosity lubricants. In contrast, valve train, piston ring assembly, and transmission clutch sliding take place under mixed or boundary lubrication conditions. The importance of different lubrication gives the different wear interacts surfaces, surface roughness at the interface, and lubricant degradation. (Michael L et al, 2004)



2.3 Lubricant

Lubrication act as an important role in reducing the mechanism of wear which eliminates the surface contact of the components. In automotive, the forms of grease are presented in motors, bearings, pumps, etc. The reaction of the lubricants affects the performance of the machine which includes high-pressure resistance, viscosity, anti-wear, and anti-corrosion resistance. Lubrication with the efficient boost gives reliability to engine and protects it from corrosion and wear. Moreover, lubricants cause a reduction in friction, temperature, wear, corrosion, and shocks. Motor oils or lubricant engine have a complex mixture which contains base and additives. The concept of the anti-wear additives is forming a thin lubricant film between contact surfaces causing the elimination of metal to metal contact. Therefore, give protection to the contact surface against abrasive, adhesive, and corrosive wear phenomena (Nazare et al., 2018).

2.4 Greases

Greases are classified according to their consistency measured by the worked infiltration stated in the National Lubricating Grease Institute (Nagendramma et al., 2015). Greases treated as semi-solid lubricants consist of soap that dispersed in synthetic oils known as a thickener. Grease gain the conspicuousness in the lubricant industry from the onset of industrial revolution machine parts. The rheological and the properties of greases influence by the constituents and microstructure of the production. MoS2 and EP additive for greases are widely used as solid lubricants with addition to graphite, PTFE, and talc. The lamellar structures of the MoS2 and graphite particles acts as a load-bearing potential which is layered with the interconnected by weak Van der Waals forces. The forces are sheared and deposited with interacting surface causing the prevention of asperities to contact. Plus, the reaction gives protection to the surface from further seizure, wear, and abrasion (Bagi et al., 2015)

2.5 Additives

The additive is a synthetic chemical substance that improves the parameter of lubricants. It boosts the existing property and suppresses the undesirable property in the base fluids. Figure 2.5 shows the grease additive level in general grease formulation. The main function of the additive is to improve the performance of the grease. There are several types of additive and each of them has a special property in the lubricant. The most common additives are:

• Anti-corrosion: -

It prevents and reduces the process of corrosion.

• Anti-wear: -

It prevents wear forming a strong surface film which reduces the contact surface to prevent heating due to extreme pressure.

• Antioxidant: -

It prevents oxidation from occurs which leads to damage and turns to admit the impurities.

• Viscosity index improver: -

It retains the viscosity of grease which will break down at high temperatures due to engine overwhelmed.



Figure 2.5: Grease anatomy

2.5.1 Type of additive

Molybdenum di sulfide (MoS2)

MoS2 is a well-known extreme pressure additive that has been largely used in greases. Dickinson et al state the properties of MoS2 are extensively attributed to the crystal structure as resulted from the crystal structure of MoS2 studied. It clearly states that a hexagonal structure of MoS2 has a sixfold symmetry layer with molecules per unit cell. The frictional behavior of MoS2 is comprehended by 'S-Mo-S' bond characteristics and the angles. In the inter-planar electron bonding, the atomic bonds are stronger in graphite. The bonding strength has six free electrons in MoS2 structure that are bound in the lattice and the existence of weak Van der Waal's forces which allows an easy shearing of adjacent 'S-S' layers.

Moreover, the polarity of the surface of MoS2 gives adsorption on the face to cleaved which endorses the adhesion of MoS2 layers onto the surface of metals. Structure of MoS2 is like that of graphene which has Mo and S in a hexagonal structure. The mechanism of friction in MoS2 conception explains by several theories' behavior of graphite. The coefficient of friction of MoS2 also depends on loading conditions that exist at the asperity contacts.

The effect of load and sliding speed on material that is un-bonded films of MoS2 was investigated which revealed the wear life of the component not fully affected by low loads and high speeds. However, at high loads and low speeds, there was a high amount of wear of the component which that the friction coefficient of MoS2 grease increases with revolutions tested. This behavior associates with the lattice structure of MoS2 as well as the formation and removal of tribofilm on the wear surface. MoS2 has a layered lattice structure that forms a protective tribofilm gives it not oriented in the direction of shearing. Thus, it would act as a pro-abrasive agent due to the presence of sharp edges and corners. At lower loads, the MoS2 would behave as an abrasive due to the inability the shear the layers which occur during the Van der Waals forces of attraction. (Salomon et al.)

Lithium Complex Grease

Greases based on lithium have better water resistance compare to sodium greases. Apart from that, it also has better high-temperature properties and excellent mechanical properties. Moreover, it has a good ability to be pumped and resistance to shear. Although lithium complex greases are more expensive to manufacture than other greases type it can give more advantages and performance compare to sodium and calcium thickeners. Lithium complex greases possess the properties of lithium soap greases which have a higher dropping point of the lithium complex grease is higher temperatures. The dropping point of the lithium complex greases is higher compare to simple lithium soap grease due to the complexing agent as a second thickener component. The modern lithium complex grease uses a shorter chain-length difunctional carboxylic acid. (T. Singh,2008)

Potassium

Mansfieal et al reported that potassium titanite largely used in the formulation of non-asbestos organic. The whisker shape potassium titanite used as substitute asbestos fibers in the material of brake friction. The binder amount of resin and potassium titanite whiskers influences the improved fade and wear resistance. Unfortunately, the application of potassium titanite causes the potential health hazard of the acicular whiskers. It extensively used in the non-steel type organic friction material which enhances friction stability at exalted temperatures. It also reduces noise and improves wear resistance of the disk.

The friction stability invented to stimulate irregular brake application in the vehicle have been investigated by using a constant interval test. The glass transition and thermal decomposition of the phenolic resin give the stabilization to the friction coefficient. This is due to the weakens glass transition with the bonding strength between the resin and the fillers in the friction material.

The results shown by Krauss test have shown that potassium titanate whiskers and splinter in the friction material have good high-temperature friction stability compared to platelets. The wear rate of the friction material is overwhelmed by the potassium titanate and the titanite. The thickness of the transfer films affected the wear resistance suggested by the examination of the surface morphology.

Sulfhydryl

Thiol group or sulfhydryl group is a compound of the organosulfur form R–SH, where R indicates as an alkyl or other organic substituent. The thiol-carboxylic adducts are effective in reducing the oxidation of the lubricant and lead corrosion. Apart from that, the lubricant compositions comprising a thiol-carboxylic adduct that react to the product of thiol and carboxylic acid derivative are disclosed. The hydroxyl group of the carboxylic acid and carboxylic acid derivative with the C1-C20 hydrocarbyl group. The lubricating composition also consists of an anti-wear agent. The lubricating compositions comprising a thiol-carboxylic adduct compose an oil of lubricating viscosity. The oils include natural and synthetic oils from hydrocracking, hydrogenation, and hydro finishing. (E.El Ashary., 2014)

E. El Ashry, E. EL-Rafey, N. Rezki et al. stated that the functionalized with thiol groups act as antioxidant additives for lubricating oils. The thiol methyl groups give an increase of antioxidant property compare to the thiol group. As the correlation on the electron-donating and accepting abilities substitute for the oxidation stability. The highest antioxidant property reaches the level of standard one in which the concentration should be 1.0% wt instead of 0.8%. The AMI calculations in the semiempirical gas-phase stated that the antioxidant power the correlation is used to investigate the character of antioxidant which is the heterocyclic additives with the structure. Due to dissolved oxygen, the additive concentration may decrease with time. Thus, it has been valuable as a useful life lubricant and the benefits of adding antioxidants for the correlation of the additive-concentration with time.

Besides the change of substituents on heterocycles affected antioxidant stability.

Polytetrafluoroethylene (PTFE)

Polytetrafluoroethylene known as PTFE is a synthetic fluoropolymer. PTFE does to a substance like water and water-contains due to adhesion to the PTFE surface is inhibited. It is widely used in various activities such as containers and pipework for reactive and corrosive chemicals as its character is very non-reactive. As a lubricant PTFE causes a reduction in friction, wear, and energy consumption of machinery. On the other hand, PTFE acts as a friction modifier in a lubricant. In grease, the PTFE use to increase the loadbearing capability. The physical reaction in between the PTFE and the metal substrate causing the thin layer of PTFE adheres to the contact surface. Mechanical fracture of the PTFE chain causing the chemical reactions induces on the surface give the highest roughness contact. Unfortunately for a prolonged load, PTFE deteriorates from the clod-flow, and leads of the wear rate increase. (Reick., 2017)

Zinc dialkyldithiophosphate (ZDDP)

One of the additives involved is sulfur-based extreme pressure which can enhance wear performance of the grease. There are several classes of sulfur carriers in greases which are sulfurized olefins/esters, dithiocarbamates, thiadiazoles, dithiophosphates, etc. Sulfur contains EP additives known as surface-active which react with metal surface forming the sacrificial tribofilm and give protection to metal from further wear. The other additives compatible with sulfur formulation are a combination with aminic antioxidants or Zinc dialkyl dithiophosphate (ZDDP) (Shah et al., 2017).

Zinc dialkyl dithiophosphate (ZDDP) widely used as an anti-wear and antioxidant additive. The ZDDP forms protective tribofilm with the hard feature and glassy in nature that is eased to manufacture and low costs. Moreover, the ZDDP give an effective AW that commonly use at low operating temperature. Zinc dialkyl dithiophosphate (ZDDP) widely used as an anti-wear and antioxidant additive. The ZDDP forms protective tribofilm with the hard feature and glassy in nature that is eased to manufacture and low costs. Moreover, the ZDDP give an effective AW that commonly use at low operating temperature.

2.6 Friction Coefficient

The production of oxidation MoS2 increases the friction coefficient between interacting surfaces. EDX map shows the presence of oxidation products on the wear surface oxidation by-products dictate the life of bonded films. Since cyclic loading shows smaller regions of tribofilm presence on the wear surface, the oxidation products formed at higher pressure and temperature led to a quicker removal of sacrificial film formation. Figure 2.6 shown the MoS2 particle of morphology and F-PTFE which give a clear view in MoS2 particles have sharp edges and corners whereas the F-PTFE particles have much smoother and rounded edges. (S. Bagi et al., 2015)



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Figure 2.6: (a) SEM images of MoS2 particles at $500\times$; (b) SEM images of F-PTFE

particles at 100×. (S.Bagi et al., 2015)



Figure 2.7: (a) Coefficient of friction (b) (c) Wear Scar Diameter

Figure 2.7 shows the comparison of the friction coefficient and dimensions of the wear scar using ASTM D2266. The graph showed the coefficient of friction (COF) between MoS2 and ZDDP + F-PTFE grease that give MoS2 increases to 0.07 and remains high for the duration test. The different result shows from the ZDDP + F-PTFE grease clearly state the COF remains low (≈ 0.03) for the first half of the test and the rises to about 0.05 and stabilizes. From the results obtained, the wear scar diameters (WSD) shown in (c) MOS2 particles twice the ZDDP and F-PTFE grease. (S.Bagi et al., 2015)
2.6.1 The Coefficient of Friction Calculation

The calculation of coefficient of friction is by the multiplication of the mean friction torque and spring constant. To measure the frictional torque in this experiment, a load cell is used. It can be expressed as:

$$T = \frac{\mu \times 3 \times W \times r}{\sqrt{6}} \tag{2.1}$$

$$\approx \mu = \frac{T \times \sqrt{6}}{3 \times W \times r} \tag{2.2}$$

 $\mu = \text{Coefficient of friction}$ T = Frictional torque (Nm) W = Applied load (N) r = Distance from the center of the contact surface on the lower balls to the axis of rotation. UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2.7 Wear Mechanism



Figure 2.8: The low magnification secondary electron images

Figure 2.8 shows the area of interest on wear mechanism with correspond to cycling load condition. This is as an evidence that the images smaller amounts of wear appeared in cyclic frequency conditions compare of cyclic loading conditions. The images display the abrasive wear mechanisms dominant in cyclic loading conditions whereas polishing wear appears to be proved in cyclic frequency conditions. The presence of tribofilm on the wear surface give protection to the surface from further wear and abrasion resulting in smaller wear scar diameters in grease. Different types of greases produce the different wear mechanism.

2.7.1 Calculation of specific wear rate

$$V = \frac{\pi h^2}{3} \times (3R - h) \tag{2.3}$$

$$h = R - \sqrt{R^2 - a^2}$$
(2.4)

- V = wear volume in, mm^3 ,
- h = height of wear scar in, mm
- R =Radius of the ball in, mm
- a = Radius of the wear scar, mm.





t = Sliding time, s

from the equation above it can be simplify as

$$K = \frac{\pi}{3t} \left(R^2 - \sqrt{R^2 + a^2} \right) \left(2R + \sqrt{R^2 + a^2} \right)$$
(2.6)

2.8 Surface Roughness

During the rolling conditions, the surface asperities appear on the body affects all the components. To investigate the effect on surface roughness adhesion and abrasion caused by the friction and wear the experimented and theoretical is performed. (Ghalme et al., 2017) J.Kang et al. states that different surface roughness values rolling against the two types of steel balls produce different resulted. The composite surface roughness contact is most influential in rolling contact fatigue (RCF) life. The effects of contaminant composition and morphology on rolling contact fatigue and wear behavior. Besides, the material properties on fatigue and wear behavior also affected. Developed the theory of hydrodynamic lubrication between the surface affect the surface roughness developed by Hamilton

The roughness of the worn surfaces is independent of the wear due the roughness has a direct relationship with the surface's capabilities in the lubrication regime. There is a main interest in the characterization of surface roughness for functionality which reveals the surface through the spatial distribution of asperities. The indicator of friction force and surface topography are COF and roughness parameters. The final variability also includes tribological behavior, contact mechanics, and material properties.



Figure 2.9: Steel ball scars topography by optical profilometer

Figure 2.9 shown before filtering the roughness calculation of the topography of the ball. From the analysis, the COF test signal and the roughness of the ball have been accomplished in the domain of time followed by the frequency analysis. The result showed the ball space parameters of roughness are correlated with COF variation as in the Table 2.1. The consistency in the autocorrelation roughness parameter shown in profile analysis based on wavelet filtering. Moreover, the correlation coefficient with the different signs is related to the material properties and behavior under dynamic load. It is advised for further research on the result of the experiment in a dynamic process. (R. Calvo et al.2017)

	A A	AISI	316L ball	Ti6	Al4V flat
Reference	Parameter	Mean	R ²	Mean	R ²
ASTM G133	COF [dimensionless]	0.53	1	0.53	1
ISO 4287	Ra [µm] - Arithmetical mean height	6.57	-0.43	14.62	-0.40
Amplitude	Rz [µm] - Mean peak-to-valley height	47.80	-0.32	65.98	-0.88
	Rp [µm] - Maximum height of the profile above the mean line	23.57	-0.13	33.82	-0.04
	Rv [µm] - Maximum depth of the profile under the mean line	24.25	-0.66	32.15	-0.89
	Rt [µm] - Maximum peak-to-valley height of the profile	48.21	-0.33	65.98	-0.88
	Rq [µm] - RMS height	8.32	-0.35	16.74	-0.44
	Rsk [µm] - Skewness	-0.09	0.35	0.35	0.93
	Rku [µm] - Kurtosis	3.34	0.40	1.89	0.30
	Rmr (c) [%] - Material component (c=1 µm under peak)	0.81	0.10	0.81	-0.64
	Rdc[µm] - Material ratio at a profile section of p=20% and q=80	% AY 13.94	-0.47	34.39	-0.20
ISO 4287	$RSm\left[\mu m\right]$ - Mean spacing between profile peaks at the mean lin	e 55.86	0.75	56.34	0.16
Spatial	RPc [peaks/mm] -Density of peaks	8.98	-0.81	8.82	-0.01
ISO 4287	R∆a [mrad] - Mean slope of the profile	0.098	-0.87	0.082	0.80
Hybrid	R∆q [mrad] - RMS slope of the profile	0.120	-0.83	0.095	0.80

Table 2.1: Roughness based on profile parameters and correlation COF

CHAPTER 3

METHODOLOGY

3.1 Overview

The objective of this study is to investigate friction and wear performance of greases with different additives. This chapter will describe the method used in this project to get the desired results. Overall operational flow is to illustrate the order to conduct the experiment as shown in Figure 3.1 Start experiment Literature Review Sample Preparation MELAKA Tribology test No COF, wear diameter Yes **Comparing Result Report Writing**

Figure 3.1: flow chart of the experiment

Experiment end

3.2 Sample Preparation

The materials are provided by the MNR Multitech Sdn Bhd which MNRg-Treat is the bio-coagulant treatment for oily wastewater. Meanwhile, MNRg-Grease refer to production of multipurpose grease from industrial waste. MNR1 is added with the molybdenum (MoS2), MNR2 is added with lithium complex grease (LCG), MNR3 is added with sulfhydryl group or thiol group (SH), and MNR4 is added with potassium (K) as shown in Figure 3.2.



3.3 Tribological test

The Four-Ball Testing Machine as shown in Fig.3.3 is functionalized to measure the properties of greases with a suitable standard. The three stationary steel balls which immerse in the grease contact with point interface of the rotating diameter steel ball under the application load. The speed of rotation, normal load, and temperature can be adjusted using an electronic controller according to the standard test schedule. The main objective of this machine is to determine the load-carrying capabilities of lubricating grease. The four-ball tester is an excellent instrument for development and quality check for the users of lubricating oils. The stability and repeatable contact can be gain by a unique configuration that gives a repeatable result. Moreover, this machine is a good R&D product due to the ability to measure the repeatable and quick results. From the test the size of the scar will be obtained and shows the ability of the lubricant to prevent wear. Poor wear presentative property indicates by the large size of wear scar while the smaller indicates superior wear preventive property.



Figure 3.3: The schematic of four ball test



Figure 3.4: 4-ball Testing Machine

Figure 3.4 shows the Ducom four-ball tester is to designate lubricants to wear preventive (WP), extreme pressure (EP), and frictional properties. The test configuration is to determine the rolling contact fatigue and shear stability behavior with appropriate attachments. The Four-Ball Tester is integrated with apparatus and controller which authorizes the measurement of normal load, speed, temperature, and frictional torque. The data is collected and displayed graphically on-line according to the standard ASTM D266. The scar diameter is measured with a measuring microscope or image procurement system. Graphs of results can be superimposed for a comparative and optional scar which use to measure the image procurement system with an image sensor permits acquiring of scar image.





Figure 3.5: (a) Power Supply (b) PC (c) Electronic Controller

The main power is switched on as shown in (a) to give power supply to the machine. Then, the switch located behind the equipment is turned on (b) as well as the Electronic Controller and the PC (c). The process needed to ensure the data collected from the experiment is gained by the PC. The electronic controller is to control the temperature, speed, torque, and the period of the experiment. For this test, the standard is ASTM D2266 as shown in table 3.1. Table 3.1 shown the parameter test in American standard while in table 3.2 the parameter test is in metric form. There are slightly different as the load for the metric form is in newton (N) and for the American standard, the load is in the unit (kgf). In this experiment, the speed is 600 rpm which needs to be set in the electronic controller of the four-ball tester.

Table 3.1:	Design	of exp	eriments
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Test	Load	Revolution	Speed	Time	Temp	Graphical representation
Protoco	(kgf)	for each	(rpm)			
l		load				
		segment				
ASTM	40	72000	1200	1 Hr	75°C	
D2266						
Cyclic	40-80	1800	1200	1 Hr	75°C	
<u> </u>		WALAYSIA 4		1	1	

Table 3.2: Testing parameter for 4 ball

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Additive	Load	Speed	Time	Temperature
a same	(N)	(rpm)	(s)	(°C)
Molybdenum	12	1 0		
Lithium Grease Complex	يكر	ى ئىك	ىيۇرىسى	او
Grease Sulfhydryl	400 KNIKAL	600 MALAYS	3600 IA MELAP	75 (A
Potassium				



Figure 3.6: (a) Ball bearing (b) Pot (c) Covers (d) Large component

The standard ball pot is set up assembly by place 3 ball bearing inside the pot. (a) Before the ball is placed in the pot it needs to be clean up with acetone. (b) Next, the small component is inserted into the ball pot to ensure the ball in place. (c) Then, the grease is placed sufficiently covers the ball bearings to the level of approximately 7ml. The grease to be touching the ball as the main point of the experiment is to increase the performance of the ball tested. (d) The pot is secured with a large component. The important step in this experiment to ensure the covers stick together with the large component so that it can be placed in the testing machine.







The ball pot assembly is locked by placing the 'ALLEN KEY SET' (a) at the top of the pot and the pot at the edge of the loading table. Then, the pot is locked by moving the key in the clockwise direction (b). the key needs to be move until the sound 'tick' appears it shows the covers fully lock with the large component. The fourth ball bearing is placed into the slot (c) (d). Before inserted the ball, it needs to have a special tool located at the edge of the testing machine to ensure the ball stick to the slot and not easy to fall.



(b)



Figure 3.8: (a) Ball pot (b) Sensor Wire (c) Load hanged

The ball pot is correctly placed on the mounting disc to ensure it is stable. Plus, it is crucial to ensure the ball pot touches the other ball after the load is applied (a). The machine provides a vertical shaft which allows the mounting to mobile the ball. Then the temperature sensor is connected to the pot as shown in figure 3.8 (b). The load is hanged until it reaches 392N (40kg) according to the ASTM 2266 (c). The loading is also obtained by the middle point fixed lever.



The 'WINCUCOM' software is open in PC to gain the data from the experiment. Then, the 'START' button is clicked on the screen. The torque is set to be 0 then the 'START' button is pressed at the electronic controller. The four-ball tester machine is integrated with a controller which permits the load, speed, temperature, and frictional torque. The data graphically display on-line and the scar diameter is measure by the microscope





4.6

(a) (b)

Figure 3.10: (a) Ball Bearing (b) Allen Key (c) WINCUCOM

After the machine stop, the load is removed as well as the temperature sensor wire and the ball pot. Then, the ball bearing is removed from the slot using the collect master. The gloves are needed when removing the temperature sensor wire and the ball pot (a). The large component from the ball pot is unlocked by pulling in the anti-clockwise direction (b). The ball bearing can be taken out and observed under the microscope. Data provided can be extracted and saved for further use (c).

3.4 Wear Scar and Surface Roughness Observation

The magnified image of the specimen after done with the wear test is obtained using the 3D non-contact profilometer. It is capable to focus on the wear track and produce a 3D profile of the magnified image. For this study, the 3D non-contact profilometer used to obtained wear scar radius and wear depth through a 3D profile of wear track.



UNIVERSITI TEKNIKAL MALAYSIA MELAKA Figure 3.11: 3D non-contact profilometer

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

This chapter will be discussed on the results from the experiment that have been conducted which contain the surface roughness, friction coefficient, and wear data. Specific calculations are performed to obtain the wear rate of the specimens. The data will be analyzed and explained occurred during the test. This chapter is divided into 5 sub-section which are section 4.2 will be explained on the experimental data of the ball. Section 4.3 focuses on the coefficient of friction and the section 4.4 on the wear rate analysis of the specimen. Lastly, 4.5 will show the surface roughness formed in MNR with different additives.

4.2 Experimental DataTI TEKNIKAL MALAYSIA MELAKA

4.2.1 Testing parameter

Additive	Load (N)	Speed (rpm)	Time (s)	Temperature (°C)
Molybdenum				
Lithium Complex Grease	400	600	3600	75
Sulfhydryl	100	000	5000	10
Potassium				

Fable 4.1:	Testing	parameter	for	4 ball
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4.3 Coefficient of Friction Analysis



4.3.1 Coefficient of Friction (COF) of Each Specimen

Figure 4.1: Coefficient of friction against sliding time with molybdenum additive



Figure 4.2: Coefficient of friction against sliding time with lithium complex grease additive



Figure 4.3: Coefficient of friction against sliding time with sulfhydryl additive



Figure 4.4: Coefficient of friction against sliding time with potassium additive



4.3.2 Comparison of COF Over Sliding Time

Figure 4.5: Variation of coefficient of friction with different types of additives

Figure 4.5 shows the result from four-ball test for four different types of grease at room temperature (24°C). The average coefficient of friction was determined from 1000s to 3000s of sliding time as shown in Figure 4.6. The highest COF was molybdenum followed by potassium, lithium complex grease and sulfhydryl. Figure 4.5 shows the summarize from figure 4.1(Molybdenum), figure 4.2 (Lithium Complex Grease), figure 4.3 (Sulfhydryl) and figure 4.4 (Potassium) using the average coefficient of friction. From the data obtained the grease with molybdenum gives the lowest COF compare to the other additive. The coefficient of friction needs to be low to achieve the best grease formulation that will reduce the friction impact.

4.3.3 Comparison of Average COF



Figure 4.6: Variation of average CoF with different type of additive

Figure 4.6 represents the coefficient of friction for the grease samples with different additives that were tested. The average coefficient of friction obtained through the test using molybdenum additives is 0.038. Lithium complex grease gives a higher value compared to the molybdenum which is 0.052. It shows a stable and constant friction response under the D2266 protocol. Sulfhydryl additive is 0.073

which exhibits higher COF compared to molybdenum and lithium complex grease additives. For the potassium additive gives a value 0.058 which is slightly lower than sulfhydryl additive but higher compared to the lithium complex grease and molybdenum. Sulfhydryl additive has the worst performance among the four tested MNR samples. From the experiments obtained, it is proved that grease containing organic additives with sulfur in their structure gives the best performance under both test protocols. On the other hand, inorganic additives that did not contain sulfur in their molecular structure formulations, exhibited poor friction performance when compared with all the samples.

4.4 Wear Analysis



4.4.1 Wear Scar Diameter

Figure 4.7: Variation of specific wear rate with different additives

From the data as seen as illustrated in figure 4.7. Wear scar diameters (WSDs) were calculated using images obtained from 3D profilometer. Figure 4.7 shows the plot for the WSDs with corresponding error bars obtained for all grease formulations and test protocols. In addition, molybdenum additive has the smallest WSD under the test protocols. The lithium complex exhibited a slight increase compared to the with molybdenum which is 0.459. While sulfhydryl additive gives a higher value, which is 0.498 while potassium additive gives a slight lower compare to the sulfhydryl but higher compare to the lithium complex grease. Sulfhydryl exhibited significantly larger wear scars under the test performance when compared with another additive. The additive which are sulfur-free additive had the worst friction and wear performance above all additive tested.

4.4.2 Specific Wear Rate

Additive	Wear Scar Diameter mm	Specific Wear Rate mm ³ /s
Molybdenum (MoS2)	0.340	1.6179
Lithium Complex Grease (LCG)	0.459	1.6471
Sulfhydryl (SH)	0.498	1.6471
Potassium (K)	0.463	1.6471

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From the calculation, the grease with molybdenum additive shows the lowest specific wear rate with a value of 1.6179 compared to other samples. The grease with LCG, SH, and K give the same amount of specific wear rate this due to the wear scar diameter that provided from the test.

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4.4.3 Surface Morphology of Wear Scar



Figure 4.8: Surface morphology of wear scar for different type of additive

From the previous study the characterize wear surface and wear mechanism to determine abrasive wear, polishing wear, and adhesive wear. From all the samples in figure 4.9, it can be seen the molybdenum additive is smaller and appear in the circular shape of wear. For lithium complex grease show more abrasive compare to molybdenum. Sulfhydryl showed small patchy tribofilm are spread and potassium showed the polishing results. Therefore, it can be concluding the grease with sulfhydryl has the worst surface roughness, wear scar diameter and coefficient of friction due to the inorganic additive did not contain sulfur in their molecular structure formulations.

4.5 Surface Roughness



Figure 4.9: Surface roughness Ra of grease with different additives

From the data obtained in figure 4.8, it shows that different results compare to the wear scar diameter and coefficient of friction. In the surface roughness Ra, potassium (K) shows the lowest surface roughness compare to the other additive which states 0.95. While the molybdenum gives a higher value (1.9) compare to lithium complex grease and potassium additives. However, grease with sulfhydryl is the highest surface roughness compared to all grease. This can be clearly explained using the surface morphology of the wear scar.

4.6 The Result Comparison

Additive		TEST RESULTS	
	Wear data	Friction data,CoF	Surface
			roughness
	Average wear scar	Taken at average of	Average of
	diameter (mm) from	1000s to 3000s	surface roughness
	3 measurements		using
			profilometer
Molybdenum	0.340	0.038	1.90
(MoS2)	CLARK I		
Lithium Complex	0.459	0.052	1.55
Grease (LCG)			
Sulfhydryl (SH)	0.498	0.073	3.59
Potassium (K)	0.463	0.058	0.95
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Table 4.3: Result Comparison from Test

The experiment shows that the grease with molybdenum additives required the lowest friction coefficient and wear scar diameter due to its characteristic as an extreme-pressure additive in greases. It is believed that sulfur-based extreme pressure enhances the wear performance of greases. It is widely used in the industry as it provides the anti-wear and extreme-pressure properties in automotive grease. (A. Shah et al. 2017) However, for the surface roughness, it gives slightly higher due to the morphology of particles with sharp edges. It behaves as a pro-abrasive agent due to the insufficient for lamellar layers shear on the wear surface. (S.Bagi., 2015)

Grease with lithium complex greases enhance a low-temperature performance which indicates the low frictional torque and excellent oil film capability. Unfortunately, it tends to exhibit low rates of base oil at low temperature which lead to surface dry. (K. Harris et al. 2017) Therefore, it is relevant the lithium complex grease (LCG) gains slightly low in friction coefficient and wear scar diameter compare to other additives. The surface roughness shows slightly lower compare to SH and MoS2 due to its surface morphology that behaves as abrasive wear. Thus, it can be proved that lithium-based give smooth efficiency grease at the higher load. (T.singh 2008)

The highest friction coefficient, wear scar diameter and surface roughness required from the grease with sulfhydryl (SH). Sulfhydryl act as a compound that adducts for an effective in reducing oxidation leads to corrosion. The dissolved oxygen reacts with the compound causing the additive concentration to decrease with time. (E.El Ashary., 2014)

Grease with potassium (K) additive give slightly higher friction coefficient and wear scar diameter compare to others. Potassium acts as the friction stability which reduces noise and improves wear resistance. However, the potassium transfer films produced not sustainable at high temperature due to it easily detached during sliding leads to poor wear resistance (O. Paper.,2008)

Chapter 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In this study, four different formulations were blended based on the additive to study the effect of the wear scar, friction performance, and surface roughness of the greases. Based on the result obtained, the grease with molybdenum additive exhibited the best performance as compared to other additive based on its behavior. Molybdenum additive has the lowest coefficient of friction at an average of 1000s to 3000s which is 0.038 compare to the other additives Lithium (0.052), potassium (0.058) and thiol (0.073). Moreover, the average wear scar diameter (mm) from the measurements also showed that molybdenum gives the lowest wear scar reading which is (0.340). The result is due to the molybdenum property of the extreme pressure additive that also prevents wear forming a strong surface film which reduces the contact surface to prevent heating. Even though, for the surface roughness, the data provided is not the lowest but still in the lower range (1.9). The sharp edges on the molybdenum particle behave as an abrasive agent. In chemical theory, it due to the insufficient forces to overcome the Van der Waals forces that help formed the tribofilms on the wear surface.

5.2 Recommendation for Future Studies

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There are several improvements and recommendations that could be made for further study to complete this research. The data obtained from this study shows the variation of results depends on the composites of the greases. However, it only emphasizes the standard of the same load. For future work, the four samples will be tested on the seizure test. From the result, the best extreme pressure additives can be selected in tribochemistry by topography by SEM techniques. In general, a series test conducted the wear-scar diameter values increase gradually up to ISL. The load increase gives a sharp rise in the wear-scar diameter value. The mixed film separates the contact surfaces and lowers the friction coefficient and wear-scar diameter values even at higher load. Therefore, further study will be conducted to observe the best additives in greases that have a low COF, WSD, and low abrasive wear of surface roughness in the seizure test.

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APPENDIX A

Appendix A: Project Gantt chart for PSM 1

	Task Name	Duration	Start	Finish	Predecessors
1	Project Selection	2 wks	Mon 9/9/19 8:00 AM	Fri 20/9/19 5:00 PM	
2	Introduction	1 wk	Mon 23/9/19 8:00 AM	Fri 27/9/19 5:00 PM	1
3	literature review	1 wk	Mon 30/9/19 8:00 AM	Fri 4/10/19 5:00 PM	2
4	Design Experiment	1 wk	Mon 7/10/19 8:00 AM	Fri 11/10/19 5:00 PM	3
5	Mini Presentation	1 wk	Mon 14/10/19 8:00 AM	Fri 18/10/19 5:00 PM	4
6	Submission Progress Report	1 wk	Mon 21/10/19 8:00 AM	Fri 25/10/19 5:00 PM	5
7	Submission Final Report	7 wks	Mon 28/10/19 8:00 AM	Fri 13/12/19 5:00 PM	6
8	PSM I Talk	2 days	Mon 23/12/19 8:00 AM	Tue 24/12/19 5:00 PM	7
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	EKM	NA.			

APPENDIX B

Specific Wear Rate 4 Types of Specimen

From the equation 4.6 the specific wear rate of the samples can be obtained with the data provided

For molybdenum (MoS2):

Diameter of ball = 12.7 mm; Wear scar diameter = 0.340 mm

$$K = \frac{\pi}{3(3600)} \left((12.7)^2 - \sqrt{(12.7)^2 + (0.340)^2} \right) \left(2(12.7) + \sqrt{(12.7)^2 + (0.340)^2} \right)$$

$$K = 1.6179 \ mm^3/s$$

For lithium complex grease (LCG):
Diameter of ball = 12.7 mm; Wear scar diameter = 0.459mm

$$K = \frac{\pi}{3(3600)} \left((12.7)^2 - \sqrt{(12.7)^2 + (0.459)^2} \right) \left(2(12.7) + \sqrt{(12.7)^2 + (0.459)^2} \right)$$

$$K = 1.6471 \ mm^3/s$$

For sulfhydryl (SH):

Diameter of ball = 12.7 mm; Wear scar diameter = 0.498

$$K = \frac{\pi}{3(3600)} \left((12.7)^2 - \sqrt{(12.7)^2 + (0.498)^2} \right) \left(2(12.7) + \sqrt{(12.7)^2 + (0.498)^2} \right)$$

 $K = 1.6471 \ mm^3/s$
For potassium (K):

Diameter of ball = 12.7 mm; Wear scar diameter = 0.463 mm

$$K = \frac{\pi}{3(3600)} \left((12.7)^2 - \sqrt{(12.7)^2 + (0.463)^2} \right) \left(2(12.7) + \sqrt{(12.7)^2 + (0.463)^2} \right)$$

 $K = 1.6471 \ mm^3/s$

