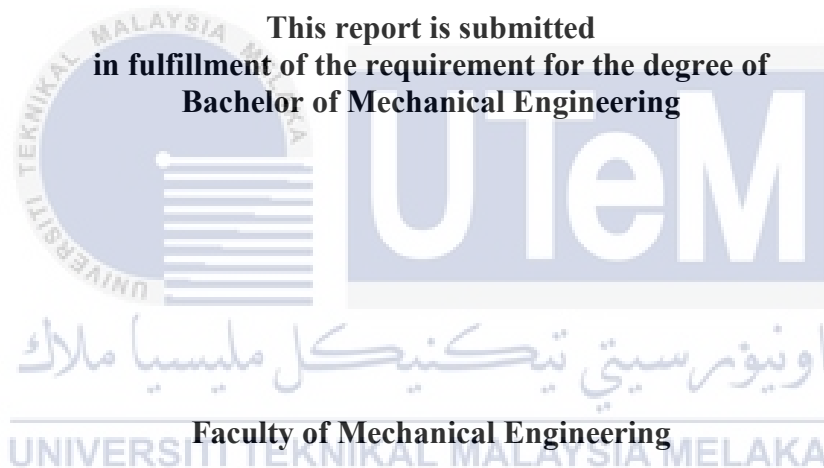


**TRIBOLOGICAL PERFORMANCE OF EXPANDED GRAPHITE AS OIL
LUBRICATING ADDITIVE**

MUHAMMAD YUSUF HIDAYAT



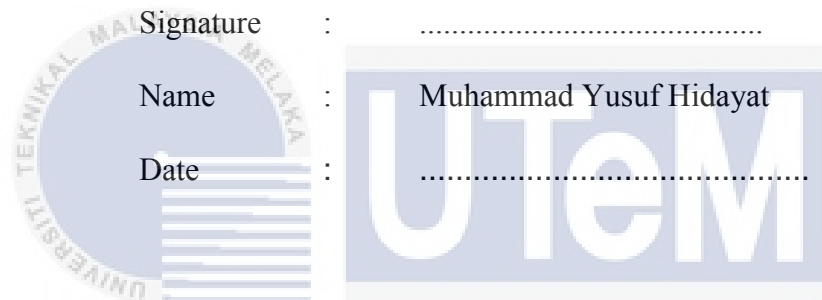
UNIVERSITI TEKNIKAL MALAYSIA MELAKA

MAY 2019

DECLARATION

I'm Muhammad Yusuf Hidayat declares that this project report entitled "Tribological Performance Of Expanded Graphite As Oil Lubricating Additive", is the result of my own work except as cited in the references

Signature	:
Name	:	Muhammad Yusuf Hidayat
Date	:



اونيورسيتي تيكنيكل مليسيا ملاك

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

APPROVAL

I hereby 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 (Hons).

Signature :

Name of Supervisor :

Date :



DEDICATION

To my beloved mother and father



ABSTRACT

This study is focusing on the effect of mineral engine oil with Expanded Graphite (EG) nanoparticles as an additive. The optimal composition of additive added into mineral oil is about 0.5% volume and it is dispersing by using a sonication technique. Then, the most optimal temperature in the Coefficient of Friction is at 75°C. The tools for the tribological testing is Four Ball Tester according ASTM D-4172 standard. The volume loss of ball is analyzing to get the value of wear volume between the engine oil with additives and non-additives. Minimum value of wear volume is found at 0% and 0.5%, both of these show the smallest wear volume in base engine oil when compared to 0.2% EG. For coefficient of friction in term of sliding time. At 75°C, engine oil with 0.5% EG has the best coefficient of friction at a stable engine running condition when compared to all engine oils against temperatures of 40°C and 120°C. Then, Wear scar observation on 0% and 0.5% EG at temperature 75°C. They show different wear patterns with parallel grooves. 0.5% EG has shallow scratches when compared to 0% EG which has more deep grooves. The result of the experimental studies has shown a potential and effective way in reducing friction and wear at the most optimum temperature by apply EG as an additive in the lubricant oil.



ABSTRAK

Kajian ini memberi tumpuan kepada kesan minyak enjin mineral dengan nanopartikel yang diperluas Grafit (EG) sebagai aditif. Komposisi aditif yang ditambah ke dalam minyak mineral adalah kira-kira 0.5% dan menyebar dengan menggunakan teknik sonication. Kemudian, suhu yang paling optimum dalam Pekali Gesekan adalah pada 75 ° C. Alat-alat untuk ujian tribological adalah Four Ball Tester menurut standard ASTM D-4172. Kehilangan jumlah bola menganalisis untuk mendapatkan nilai volume pakai antara minyak enjin dengan bahan tambahan dan bahan tambahan. Nilai minima voltan pakai didapati pada 0% dan 0.5%, kedua-duanya menunjukkan volum haus terkecil dalam minyak enjin asas apabila dibandingkan dengan 0.2% EG. Untuk pekali geseran dari segi masa gelongsor. Pada 75 ° C, minyak enjin dengan 0.5% EG mempunyai koefisien geseran terbaik pada keadaan enjin yang stabil apabila dibandingkan dengan semua minyak enjin terhadap suhu 40 ° C dan 120 ° C. Kemudian, Gunakan pemerhatian luka pada 0% dan 0.5% EG pada suhu 75 ° C. Mereka menunjukkan corak memakai yang berbeza dengan alur selari. 0.5% EG mempunyai calar cetek apabila dibandingkan dengan 0% EG yang mempunyai alur yang lebih dalam. Hasil kajian eksperimen menunjukkan cara yang berpotensi dan berkesan dalam mengurangkan geseran dan dipakai pada suhu yang paling optimum dengan memohon EG sebagai tambahan dalam pelincir minyak.

اوتیورسیتی تیکنیکل ملیسیا ملاک

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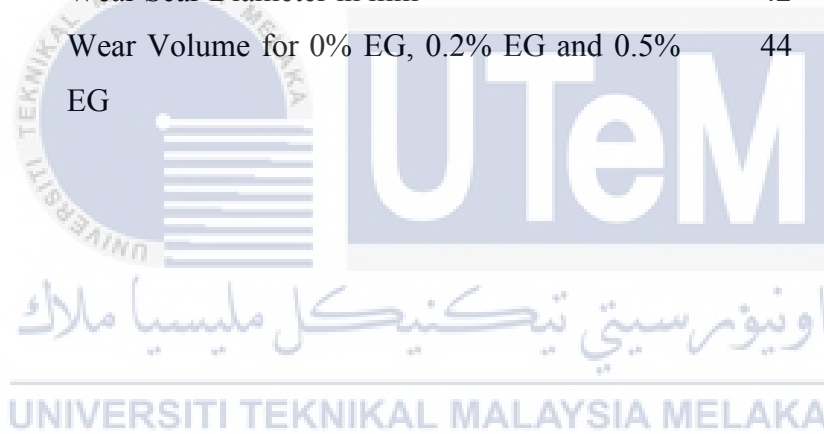
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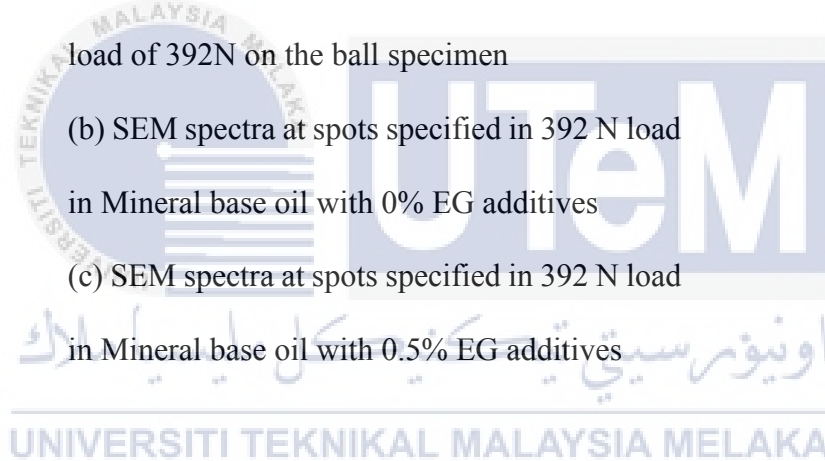
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LIST OF ABBREVIATIONS

EG	Expanded Graphite
ASTM	American Standard for Testing and Materials
SEM	Scanning Electron Microscope
CuO	Copper Oxide
TiO ₂	Titanium Dioxide
SF	Sulphur Free
NBR	Acrylonitrile-Butadiene Rubber
FLG	Few Layer Graphene
MLG	Multi-Layer Graphene
GNP	Graphene Nanoplatelets
rGO	Reduced Graphene Oxide
GO	Graphene Oxide
2D	Two-Dimensional
SAE	Society of Automotive Engineers
UTeM	University of Technical Malaysia Melaka
SEM	Scanning Electron Microscope
COF	Coefficient of Friction
EDX	Energy Dispersive X-ray
WSD	Wear Scar Diameter
TBN	Total Base Number
PC	Personal Computer
AISI	American Iron and Steel Institute

LIST OF SYMBOL

TPa	=	Terapascal
kg	=	Kilogram
g	=	Gram
RM	=	Malaysian Ringgit
ml	=	Milliliter
L	=	Liter
N	=	Newton
m	=	Meter
cm	=	Centimeter
mm	=	Millimeter
μm	=	Micrometer
Nm	=	Newton Meter
W	=	Watt
v	=	Volt
s	=	Second
HRC	=	Hardness Rockwell Scale
rpm	=	Rotation Per Minute
/	=	Per
K	=	Kelvin
°C	=	Degree Celsius
%	=	Percent

CHAPTER 1

INTRODUCTION

1.1 Background

Graphene is a new carbon nanomaterial used worldwide due to its amazing properties such as high surface area, high thermal conductivity, affluent material and high performance with low quantity of material [1]. Despite the fact that graphene requires many kind of studies, Expanded Graphite (EG) which is a highly important form of graphene is examined as a major applicant material used in mineral-based lubricant because of its properties of easy shear capability. Furthermore, graphene plays a very important part in reducing frictional behaviour and improving anti wear ability at optimal concentration when added to paraffin oil [1]. It is a great benefit in the automotive sector for the performance in strengthen, lighten and reduce noise of internal combustion engine such as piston assembly, valve train and bearings. Combination between engine oil with additive oil helps improve in lowering the mechanical loses by 10% to reduce fuel consumption [2]. Graphene is an individual layer of graphite which is used in forming a tribofilm or boundary between sliding contact interfaces and create high shield coating that prevents from corrosion and rusting. Based on previous statement, a good expectation can be obtain if graphite and mineral oil is blended together because the properties of graphite will intensify the lubrication effect. However, the disadvantages of this method is the price of graphite which cost RM 450 per 100 ml. It contributes 33-40 % of tribological properties including the study and application

of fundamental friction, lubrication and wear. Thus, it is shown that it is a serious issue to investigate on performance material of expanded graphite in Mineral Oil.

From previous research, the viscosity, thermal conductivity and flash point can be improved by nanoadditives dispersing. It also increase technical properties of the main component and pour point dramatically [2]. Therefore studies on the performance of expanded graphite (EG) in a mineral oil-based product has become a famous and critical issue. Base mineral oil when blended with nanoparticle improved the tribological characteristic by composition, shape, concentration, grain size and dispersion stabilization [2]. Therefore, the addition of different concentration of EGs should be able to enhance the tribological properties.

Utilizing wear by variation of temperature with same critical load method, the tribological performance of additive in mineral-based lubricant can be tested by using four-ball testing machine. Result can be observe from the size of the wear scar, optimum additive percentage and operating temperature. The four-ball test is one of the techniques in analysing reactions between solid contact and it is effective to improve anti-friction behaviour, anti-wear characteristic and extreme pressure properties of lubricants. Based on the result, the test method can identify not only the property of lubricant but also the additional lubricating particle such as graphene.

1.2 Problem Statement

In recent years, lubrication is a very important material asset in a mechanical system [3]. Lubricant is used in engine to develop better performance as the engine runs. The performance of a lubricant extremely depends on the additives it requires. Mineral oil based lubricant has high friction coefficient and energy losses due to friction especially in boundary regime or high load. Adding the additive nanoparticles could prevent high contact pressure between the friction surfaces. Thus, it is expected to improve wear and friction coefficient in boundary region and also extend the lifespan of an engine.

Therefore, this study investigates the expanded graphite concentration percentage in mineral oil with expectation of improving the tribological properties. Furthermore, this study will be interesting if the optimum result is gain at normal engine running temperature which is 75°C. This is because overall life cycle of an engine are in this state. To achieve the objective of the study, several methods will be used such as analysis of wear mode, wear scar diameter and steel ball profile.

1.3 Objective

The main objectives of this research are as follows:

- i. To study the new potential improvements for friction and wear of Expanded Graphite (EG) in mineral based lubricant.
- ii. To determine the optimum percentage of Expanded Graphite in Mineral based lubricant for friction and wear of steel and steel contact.

1.4 Scope of Project

The scopes that focuses on this project are:

- i. Study the varying concentration of EG with varying of temperatures.
- ii. Test using 4-ball Tester under ASTM D 4172 standard with parameter speed 600 rpm for 1 hour with load 392N.
- iii. Test at 40°C, 75°C and 120°C with load 392N.
- iv. Analyze the result using Scanning Electron Microscope (SEM) to observe the wear morphology.



CHAPTER 2

LITERATURE REVIEW

2.1 Lubrication Theory

Nowadays, lubricants are usually introduced as a substance between two moving surfaces to reduce friction and wear properties. Lubricants act as protective film that coats and protects two contact surfaces. It also smoothens out the surface by filling up the gaps of the rough asperities [4] as well as dissolving minute particles and distributing heat. Figure 2.1 shows the presence of lubrication between two surfaces in contact. Furthermore, lubricants can be used to improve the oxidation resistance which helps prevent rust and corrosion. It can also be used to control and dissolve contamination such as reaction products, wear particles and other debris. When two surfaces are moving, heat is generated due to friction. An application of a protective film in between the moving surface will reduce friction and heat generation. Thus, achieving wear reduction.

Based on previous research in various lubrication systems including gas lubrication, solid lubrication, and liquid lubrication, early civilisations used gypsum, water lubrication, and animal fat as a method to reduce friction [5]. Then, civilisations within Middle East began incorporating oil as a method of lubrication, which then made it as part as their culture and tradition [5]. This inclusion of lubrication as part of society existed centuries prior to the invention of mechanical engines. Lubricant application in daily life started from the mid-1400s, and it was only starting in the mid-17th century that petroleum-based lubricants were recognised for the reduction of friction and wear [6].

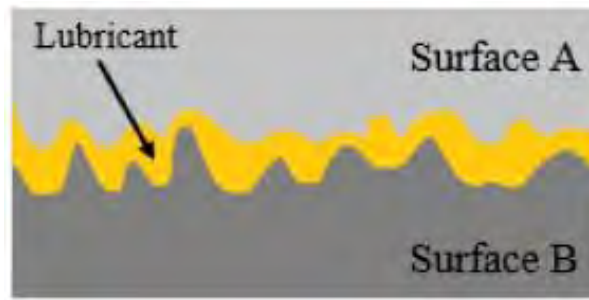


Figure 2.1: Lubrication between two surfaces in contact

2.2 Type of lubricants

There are various types of lubricant available in the market nowadays such as liquid, gas, solid and semi-solid lubricant.

2.2.1 Liquid lubricant

A liquid lubricant provides special properties such as wear replenishment, low mechanical noise, and the ability to remove wear debris [4]. Liquid lubricants can decompose or oxidise under extremely high temperature and would evaporate under extreme pressure conditions [5]. Liquid lubricants also can be useful as a cooling function and surface protection. Liquid lubricant forms a film in the hydrodynamic and hydrostatic regime. In hydrodynamic regime, the fluid enters the surface by the action of the bearing. Different from hydrodynamic, the fluid is conducted by under pressure from an external source. The bearings are made from hard plastics by rubber materials due to the performance of absorbing vibration, reduce the noise and impact of a system [6]. The lubricant application in the bearing cavity is the main factor to a long lasting surface.

It is also stated that rubber materials with the combination of water is the perfect combination for bearing systems. When the rotation occurs between the shaft and bearing, it is creating a layer of water. This is because of longitudinal grooves that followed by water and radial moves at the middle of the contacting surfaces in a thin film. Under boundary

lubrication conditions, a protective film on the rubbing surface plays an important part on its development as well as managing the wear behaviour [7]. Liquid lubricants are categorised based on the origin, either it was extracted from vegetables, animal fats, mineral oils, or of synthetic origins.

i. **Animal and vegetable oils**

Animal and vegetable lubricating oils are constant oils because they do not evaporate unless they decompose. It is produced by the fatty acids and alcohol compounds in animals. It is usually added to mineral oils to improve film formation.

ii. **Mineral oils**

Mineral oils are the most favourable and economically viable category of liquid lubricant that's been extracted from crude oils. It is stable under service conditions which formed based on carbon and hydrogen, Furthermore, it also contains sulphur, oxygen and nitrogen.

iii. **Synthetic oils**

Synthetic lubricating oils are made up of short and uniform molecules of carbon. This provides the durability to reduce heat and pressure. It is applied when mineral oils are inadequate.

2.2.2 Gas Lubricant

This type of lubricant use gases to lubricate bearings. It is low in viscosity, non-polluting, long lasting and small in frictional loss [4] .Gas lubricants usually performs in aerodynamic or aerostatic regimes at high speed and temperature. Many aircraft industries have utilized this kind of lubricant due to the constant chemical properties over the various ranges of temperature. This lubricant is able to work at high and low

temperatures which will increase the efficiency and reduce cost. Moreover, gas lubricants are reliable due to the appearance of a gas film between the bearing and the shaft to protect from wear [5]. The absence of sealing and contamination problems can also be avoided by the application of gas lubricant.

In fact, gas-lubricated bearing provide the best benefit as it is frictionless, silent, and vibration free. Gas bearings are capable of surviving through extreme conditions for wide surface velocities and are able to reduce the friction coefficient at higher load, sliding speed and sliding distance [4].

2.2.3 Solid Lubricant

Like any other lubricants, solid lubricant provide a thin layer of protection between two surfaces, albeit in powder form instead of liquid. The objective is to develop continuous adherent of soft or hard film in between surfaces which can be applied through mechanical or physical processes. It is applied on one surface before making contact with another.

Solid lubricants are required to reduce the amount of friction and wear under extreme conditions such as critical temperatures, high amounts of dust, and corrosive environment [6]. In addition, solid lubricants are more effective than other lubricants at discontinuous loading, high speeds and high loads in the presence of boundary and mixed regimes. Solid lubricants are generally classified into three different classifications: structural, mechanical and chemical lubricants.

- i. Structural properties: comprises layers of lattice-like structures which function as a friction reduction agent. Figure 2.2 shows the presence of layers lattice structures.

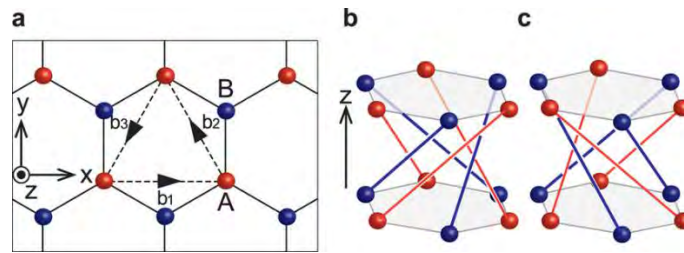


Figure 2.2: Layers lattice structures

- ii. Mechanical properties: consists of self-lubricating organic compounds like thermoplastic and thermoset. Figure 2.3 shows the presence of organic compounds like thermoplastic and thermoset.

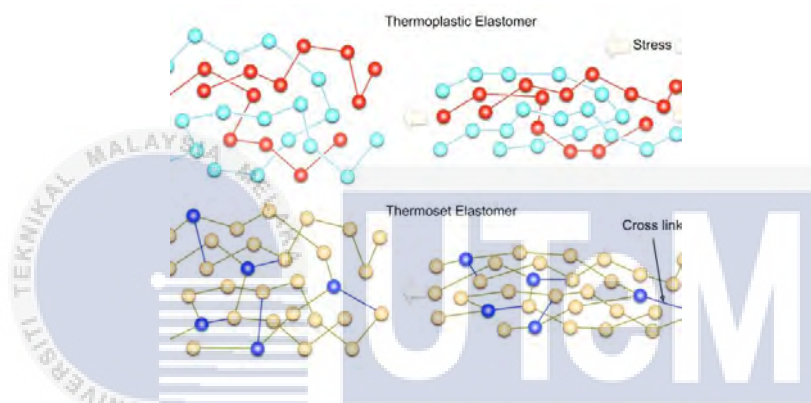


Figure 2.3: Organic compounds like thermoplastic and thermoset

- iii. Chemical properties: Established by chemical or electrochemical behaviour on the surface of metal and glasses. Usually lubrication of metal-forming reactive soap such as palmitic acid. Figure 2.4 shows the example of chemical properties in palmitic acid.

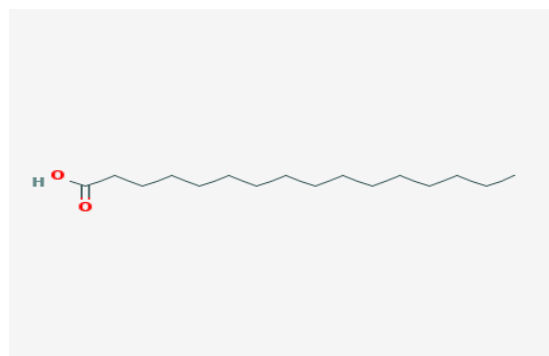


Figure 2.4: Example of chemical properties in palmitic acid

2.2.4 Semi Solid Lubricant

Semi-solid lubricants called grease are used in bearings for the purpose of lubrication. Semi-solid lubricants usually contain base oil, additives, and thickener which provides valuable properties like low friction, low shear strength and adherent film formation on the surface contact area. It is introduced as a safe technology in managing desirable cutting force, cutting temperature, tool vibration and tool wear [7]. The application of semi-solid lubricants is the heat transfer in evaporative mode which is more efficient compared to regular heat transfer in wet turning. Semi-solid lubricants are able to perform as a coolant or lubricant during turning occasion in the contact region. In addition, greases are more effective in terms of high-pressure application and cutting of metal than other lubricants.

2.3 Wear Mode

Nowadays, wear can be known as an elimination of material from a surface due to the interaction between mating surfaces. Due to this, almost all machines lose their reliability and durability. The less wear occur upon a surface, the more reliable a machine would be. Research have been done regarding wear modes and explanation of wear mechanism based on those wear modes. There are several types of wear mode, such as adhesive, abrasive, surface fatigue and corrosive wear. Figure 2.5 shows the four wear modes.

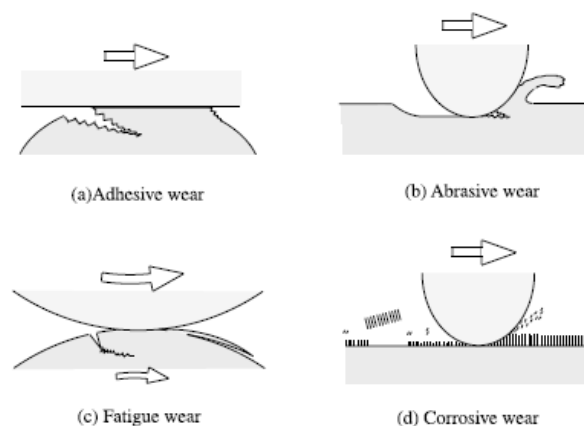


Figure 2.5: Illustration of (a) adhesive wear (b) abrasive wear, (c) fatigue, (d) corrosive [7]

2.3.1 Abrasive Wear

Abrasive wear occurs under sliding contact due to the contamination of particle. There are many characteristics of abrasive wear including low stress, high stress, gouging, and polishing. Low stress has a tendency to scratch as it is abrasive and acts as the main damage in mechanical machines. Then, high stress is a special form of abrasion which results in scratching, plastic deformation on surfaces and hole formation from impressed particles. The damage that occurs is more severe than low-stress abrasion. Gouging is the removal of material by the repetition of compression loading of a hard material such as rocks with a smoother surface, usually a metal [8]. This kind of abrasion probably together with both low and high stress. In general, Polishing wear is to smoothen and brighten a surface. This act removes materials from a surface by the application of rubbing with others solids, albeit within conditions that the material is removed without fracture, scratching or plastic deformation of the surface.

2.3.2 Adhesive Wear

Adhesive wear is the material wear that occurs due to the bonding strength between contacting solid surfaces. Similar to friction, two solids have a tendency of bonding with each other if both surfaces are closely contacting.

2.3.3 Corrosive Wear

This type of wear takes place based on oxidative reaction when the oxide layer appears between two contacting surfaces. As the impact, the surface that caused by oxide layer becomes very weak and finally release a corrosion product.

2.3.4 Fatigue Wear

Fatigue wear is generated through multiple cycles of contact stress and by the way particles are coming into direct contact with a solid surface. The wear mechanism depends

on the low or high contact cycle. The higher the contact cycle, the faster the rate of wear mechanism occurs.

2.4 Typical Stages of Wear

The wear process will go through several stages within sliding result. There are three different stages which includes initial, steady state, and catastrophic wear stage. Figure 2.6 shows illustration of the wear stage.

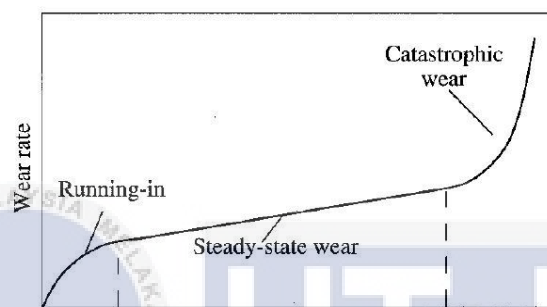


Figure 2.6: Illustration of wear stage

2.4.1 Initial Wear Stage

The initial or usually called running in wear stage is the build up to a steady state. This stage is very significant for sliding systems, gears and bearing mechanisms. From the journal, it is observed that during this stage the wear rate is high due to the small contact region and can cause high contact pressure in addition to the asperities between the surfaces [9]. When the asperities are being removed by the wear, more asperities come into the surface which leads to the reduction of contact pressure.

2.4.2 Steady State Wear Stage

This steady state region occurs in a steady wear rate. During this region, wear rates become low and stable in friction because the surface has already matched with the

asperities. The adhesive and abrasive wear also take place at this stage. Furthermore, this stage relies on velocities, temperatures, and load.

2.4.3 Catastrophic Wear Stage

This stage occurs when a severe surface failure takes place and components fail due to excessive loading and mechanical loosening.

2.5 Nanoparticle Additives

Nowadays, using nanoparticles as lubricating oil additives have gained heightened attention in tribological properties. Nanoparticles tribology is the study of lubricating oil properties and surface contacts at Nano stage. The unique characteristic of nanoparticles includes its minute size, shape, and concentration from the aspect of anti-wear, friction reduction and load carrying capacity [10].

The previous study focused on nanoparticle additives and investigated the tribological behaviour of CuO, TiO₂, and Nano-Diamond at 0.1% weight concentration. They observed that CuO exhibit excellent friction and wear reduction. It was also observed that when CuO was mixed to SF oil and Base oil, it reduced friction by 18.4 and 5.8% [10].

The other previous study used different loads on the tribological behaviour of paraffin mineral oil and deodorised palm oil. They found that palm oil showed reduction of friction coefficient under all tests loads. However, palm oil produces larger wear scar compared to mineral oil [3].

2.6 Nanoparticle Lubrication Mechanism

The investigation of the lubrication mechanism is a crucial parameter to understand the tribology of nanoparticles. Many researchers have introduced the mechanism to explain the lubrication enhancement of nanoparticle. The mechanism includes the mending effect, rolling effect, protective film formation and polishing effect that are shown in Figure 2.7.

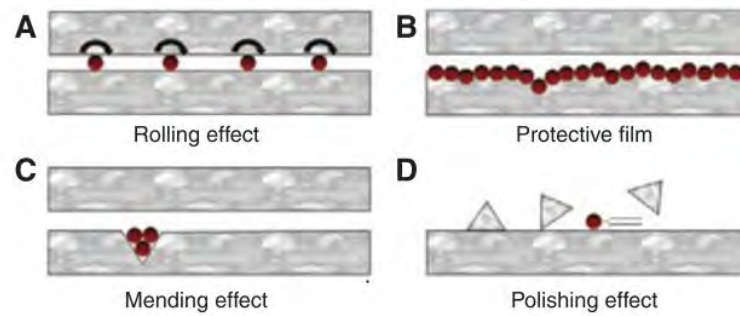


Figure 2.7: Illustration of lubrication mechanism [2]

2.6.1 Mending Effect

The mending effect is also called self-repairing effect is characterized by nanoparticles deposit on the interacting surfaces and responsible for the loss of mass. During this effect, the nanoparticles in the lubrication are capable to fill scars and grooves on the friction surface [11].

2.6.2 Rolling Effect

The spherical nanoparticles function like tiny ball bearings between two friction surfaces and roll into the contact area. This effect occurs in a tribo-pair system under low load condition between the shearing surfaces. During this effect, it maintains the shape and rigidity of the nanoparticles [12].

2.6.3 Protective Film Formation

A protective film or tribo-film is applied on the interacting surface. This film formation caused by the reaction between the additives and the treated material under origin environment.

2.6.4 Polishing Effect

The polishing effect is also called smoothing effect and act as reservoirs of solid lubricants within the contact surface to fill up the gaps of the rough asperities.

2.7 Graphene

Graphene is a type of material that made from one single layer of carbon that has been arranged in a hexagonal honeycomb lattice [13]. Graphene can be mixed with any other components consisting of metal and gases to produce all kinds of materials with various outstanding properties and essential in some other method [14]. Nowadays graphene is used as a greater alternative to conventional materials as it is lightweight, thin, flexible, conducts electricity, and improves mechanical and electrical properties of a product. There are various types of graphene based on their matrix type. These are known based on the number of layers and their natural behaviour. These consist of Few-layer Graphene (FLG), Multi-layer Graphene (MLG), Graphene NanoPlatelets (GNP), Reduced Graphene Oxide (rGO), Graphene Oxide (GO) and Expanded Graphite (EG). Table 2.1 showed properties of Graphene.

Table 2.1: Properties of Graphene [13]

Properties	Values
Electron Mobility (at room temperature) ($\text{cm}^2/(\text{V}\cdot\text{s})$)	$>2 \times 10^5$
Young's Modulus (TPa)	1
High thermal conductivity ($\text{W}/(\text{m}\cdot\text{K})$)	5300
Optical absorption (%)	3
Specific surface area (m^2/g)	2630

2.7.1 Few-Layer Graphene (FLG)

Few-layer graphene consist of two to five number of stacked layers of graphene. They are stacked based on their lateral dimension. These materials are widely used in the production of sensors, batteries, and coatings in Nanoelectronics [15].

2.7.2 Multi-Layer Graphene (MLG)

The main reason in producing the multi-layer graphene is to attach it with a coating substance or freestanding flake. Similar to FLG but with a greater deviation of the layer, ranging from two to ten number of layers [15]. It is found that it is highly beneficial in lubrication and can act as an additive material to plastic and composites.

2.7.3 Graphene Nanoplatelets (GNP)

In general, the strength and stiffness of graphene nanoplatelets can be attained due to the unique size and morphology of the material. Graphene nanoplatelets are made from 2D graphite with the lateral dimension thickness less than a hundred nm [15]. Besides that, nanoplatelets have remarkable electrical and thermal conductors because of its pure graphitic composition.

2.7.4 Reduced Graphene Oxide (rGO)

Reduced Graphene Oxide can be gained from the reduction of Graphene Oxide by minimising its oxygen concentration via chemical, thermal and microwave [15]. Reduced graphene oxide is more stable compared to GO and can transform within conductance.

2.7.5 Graphene Oxide (GO)

Graphene oxide is synthesised through oxidation of graphite powder via improved tour method [16]. The main reason for nanotribological applications is because graphene oxide has mechanical strength, tensile stress, and thermal conductivity, higher in aspect ratio and not dangerous for the environment. It is also effective for friction and wear reduction [17].

2.7.6 Expanded Graphite (EG)

Expanded graphite is a type of material that consists of low density carbon. It has a series of unique properties such as developed specific surface, binder free pressing capacity, stability to aggressive media and low thermal conductivity [18]. The material shown in Figure 2.8.

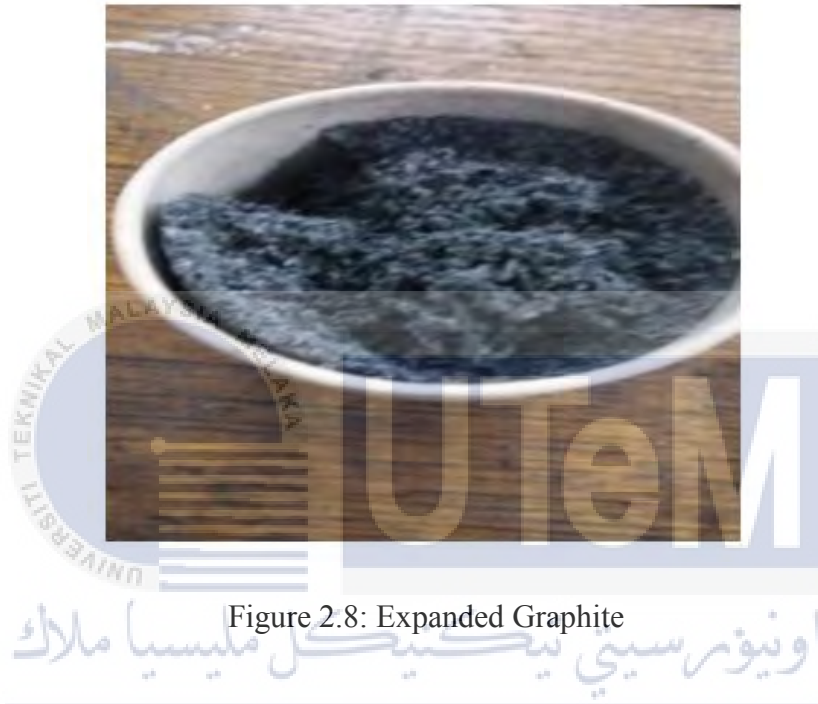


Figure 2.8: Expanded Graphite

2.8 Expanded Graphite (EG) on Tribological Aspect

Nowadays, Expanded Graphite has caught many intentions among the researches as it has high potentials for tribological properties. The main reason for nanotribological applications is because expanded graphite has mechanical strength, tensile stress, and thermal conductivity and higher in aspect ratio. It is also effective for friction and wear reduction[19].

Basically, expanded graphite is synthesised through intercalation compound of graphite that expands or exfoliates via thermal method [20]. Many studies have researched potentials and benefits of expanded as it can help to reduce wear coefficient because mostly

EG are used in airtight materials, military materials, absorbs oil, high power batteries, electrodes and flame retardant [20]. For example, an experiment has been conducted to understand lubrication effect of EG for polyimide-based composites. They found that EG improved anti-wear abilities and the reduction of friction coefficient by 0.135 [21].

Besides that, other previous study used EG mixed with acrylonitrile-butadiene rubber (NBR) nanocomposites compared to EG mix with NBR micro-composites. It is shown that the wear of polishing surface has been reduced because of EG dispersion [19]. The tribological trend of the loads effect on the friction coefficient as shown in Figure 2.9.

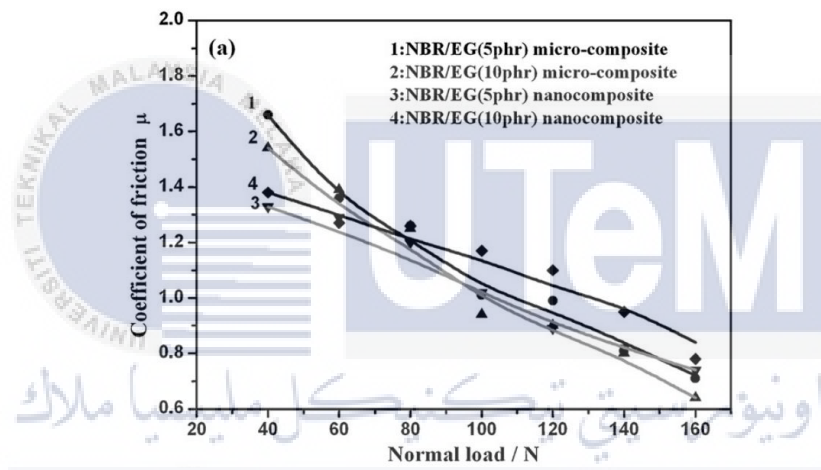


Figure 2.9: Tribological trend of the loads effect on the friction coefficient[19]

Therefore, this study investigates the expanded graphite concentration percentage in mineral oil with expectation of improving the tribological properties. To achieve the objective of the study, several methods will be used such as analysis of wear mode, wear scar diameter and steel ball profile.

CHAPTER 3

METHODOLOGY

3.1 Introduction

In this chapter, there are several actions that needed to be carried out to achieve the objective in this study. The methodology used in this study will be illustrated with more details in order to gain the optimum result of tribological properties on different types of mineral oil with additives and non-additives. The flowchart will show the overall stage of the process. This study begins by preparing different percentage of lubrication additives. Then, tribological experiment is conducted to investigate the friction and wear of steel against steel contact in different percentage of lubrication and additives.



3.2 General Process

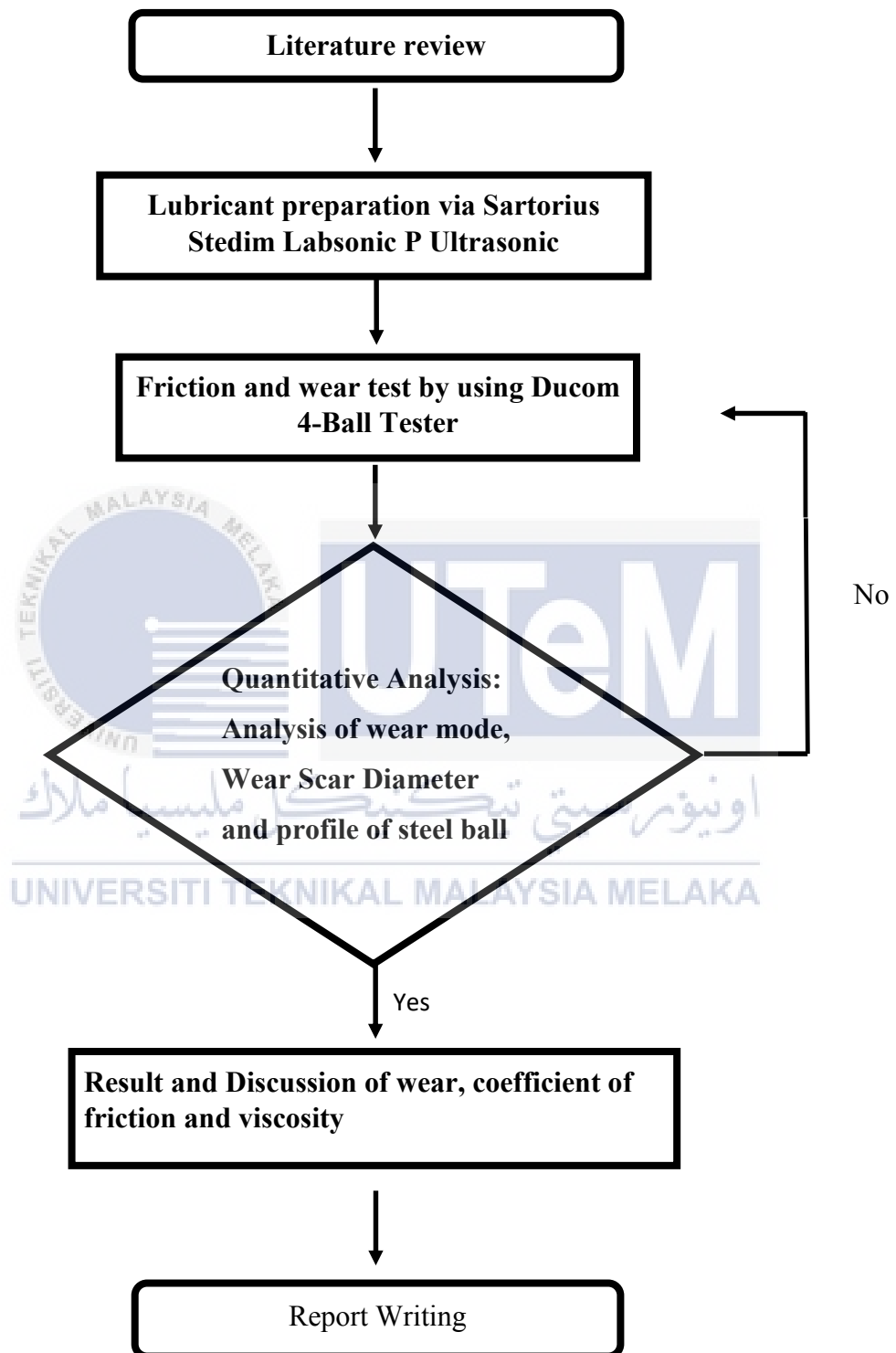


Figure 3.1: Flow Chart for FYP

Figure 3.1 shows the overall process of the study. The first stage is finding the information on EG additive and reviewing the related data from the journal, article, book, websites and others materials. By getting the information from all sources, the process related to tribological properties on friction and wear can be determined.

The significant stage in this experiment is second stage which is the sample preparation process. This stage involves a calculation of mixture additives in Shell Rimula R4X SAE 15W-40 mineral oil. The calculation and determination includes 0.5% and 0.2% of expanded graphite (EG). The mineral oil with the EG additive of 0.5% and 0.2% will be compared with the original one which is 0% additive in terms of tribological properties. Sample preparation will be conducted in tribology laboratory at Faculty of Mechanical Engineering, University of Technical Malaysia Melaka, UTeM.

After that, a 4-Ball Tester will be carried out to determine friction and wear through experiment. The studies on friction and wear using 3 different percentage of additives in the mineral oil. The temperature is tested at 40°C, 75°C and 120°C respectively. The speed and the load are the same for each test which is 600 rpm at 390 N respectively. Finally, a comparison concerning the friction and wear of different percentage EG additive test will be made. Based on the results from the data tabulation of friction and wear, the best mixture of engine oil additive will be determined in terms of low friction and wear concentration.

The final stage is to propose the wear mode of steel and steel contact in different percentage of EG. This can begin only if the tribological testing succeeded or else the tribological testing needs to be repeated once again. In order to obtain the wear mode of different percentage of EG, a surface analysis will be carried out through Scanning Electron Microscope (SEM) test. Moreover, the viscosity on friction and wear of different percentage of EG also will be investigated. This performs as a significant element of tribological properties in this experiment.

Figure 3.2 shows Gantt chart for PSM 1. It will encompass from the background of study until the preliminary results. The planning to finish the progress work will be taking about 15 weeks. Then, Figure 3.3 shows Gantt chart for PSM 2. It will cover the sample preparation, experimentation results and focus on final report writing. The expected time taken will need 14 weeks to finish.

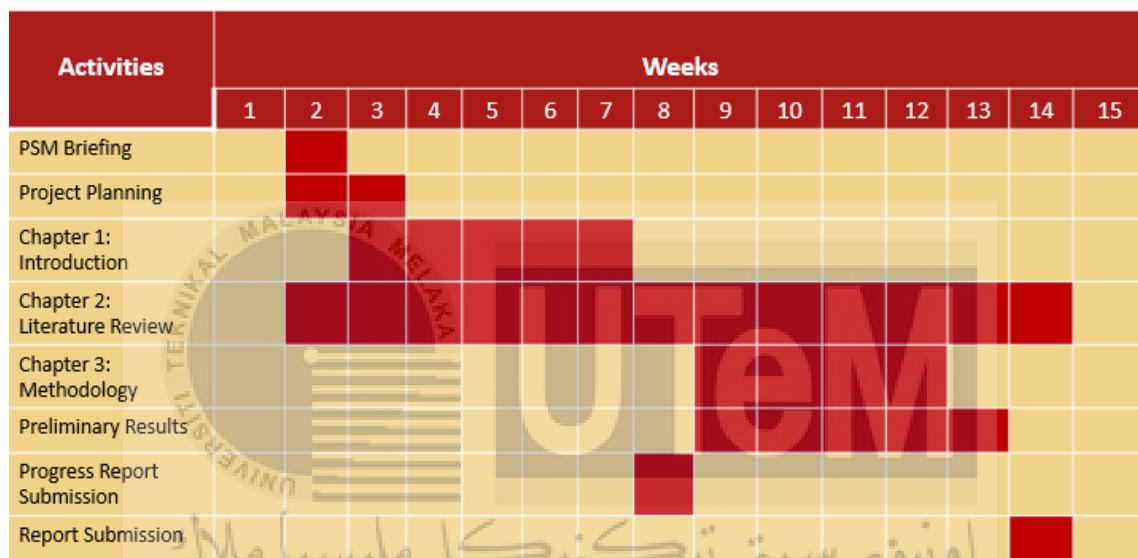


Figure 3.2: Gantt Chart for PSM 1

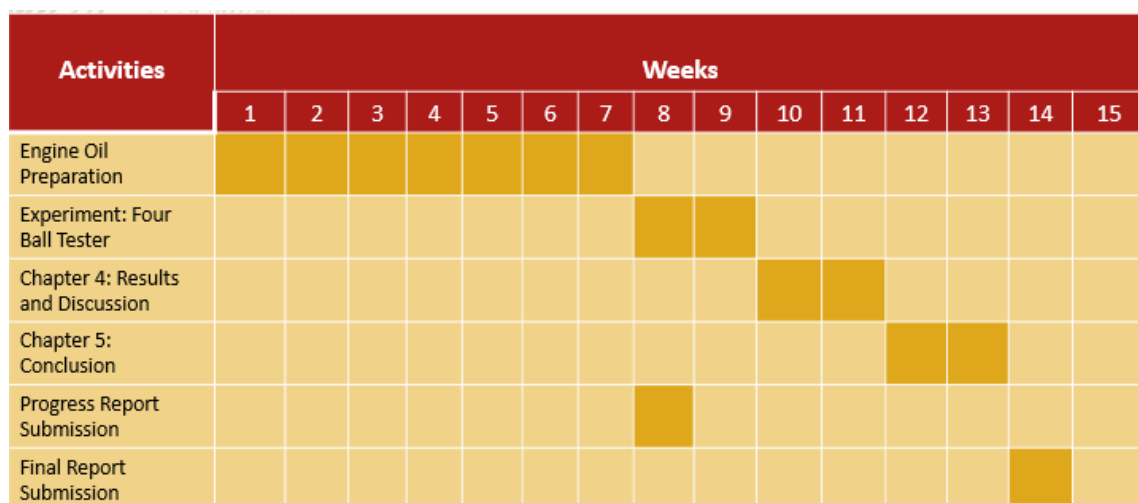


Figure 3.3: Gantt Chart for PSM 2

3.3 Experimental Methodology

Prior to the testing process, several preliminary action and various preparation exercise has been carried out to make the testing become successful.

3.3.1 Test Equipment and Specimens

The wear test was executed with a rotating friction under specified load, speed, and temperature machine via Ducom 4-Ball Tester. An experiment for lubricating oil was conducted according to ASTM D-4172. The wear test for four-ball was used to determine the friction and wear characteristic between different concentrations of EG additive. The test ball are made of AISI 52100 hardened steel with 12.77 mm diameter with 0.03 μm surface roughness and hardness value 60-65 HRC.

3.3.2 Raw Material Preparation

Mineral oil Shell Rimula R4X SAE 15W-40 was used as the base oil material that will be studied in this research. It is a flammable liquid that contains a special combination of additives and chemical to improve engine and oil durability. Table 3.1 presents some of their physical properties. In this study, the mineral oil with the EG additive of 0.5% and 0.2% will be compared with the original one which is 0% additive.

Table 3.1: Physical Properties of Base Oils

Base Oil	Density at 15°C (kg/l)	Kinematic Viscosity (mm ² /s)	Viscosity index
Shell Rimula R4X SAE 15W-40	0.88	At 40°C 109 At 100°C 14.77	139

3.3.2.1 Material Selection

For this research, the additive that's going to be used is Expanded Graphite (EG). EG is used in this project because has mechanical strength and thermal conductivity. In fact, it is also effective for friction and wears reduction. So, Mineral oil and EG will be mix together in order to determine the optimum percentage of EG in mineral oil for friction and wear of steel and steel contact. The composition of mineral oil must be dispersed with 0.2% and 0.5 % of Expanded Graphite. This forms a protective film agent. Figure 3.4 shows the sample of 0.2% EG additives in mineral oil. Then, the determination of 0.2% and 0.5 % EG is calculated as below:

Volume of solvent = 100 ml

Density of EG = 2.2 g/ml

0.2% EG Volume = mass (g)/2.2/100 ml x 100 = 0.44 g

0.5% EG Volume = mass (g)/2.2/100 ml x 100 = 1.1 g



Figure 3.4: 0.2% EG additives in mineral oil

Viscosity is one of the important properties of lubricating oil and it determines the load carrying capability as well as how easily it circulates by using HK-1005 Kinematical viscosity apparatus. It is used to test liquid petroleum products, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass

capillary viscometer. The kinematic viscosity of 0.2% and 0.5% can be calculated as shown below:

Determination of kinematic viscosity, ν for Shell Rimula R4X SAE 15W-40 with EG Percentage

Temperature = 40°C (fixed to all applied concentration)

Constant at 40°C = 0.45957 (fixed to all applied concentration)

Inner Diameter = 1.55 mm (fixed to all applied concentration)

i. EG concentration = 0.2%

Time required for the oil to pass from mark E to mark F = 254 s

Kinematic Viscosity, $\nu = 254 \times 0.45957$

$$= 116.73 \frac{\text{mm}^2}{\text{s}}$$

ii. EG concentration = 0.5%

Time required for the oil to pass from mark E to mark F = 248 s

Kinematic Viscosity, $\nu = 248 \times 0.45957$

$$= 113.97 \frac{\text{mm}^2}{\text{s}}$$

3.3.3 Test Parameter

There are about nine (9) test involved in this study of wear test condition which every of the test have different parameter. Table 3.2 shows the parameter to evaluate the tribological performance of EG as the lubricating oil additive.

Table 3.2: Wear Test Conditions

Mixing EG with mineral oil concentration (% Volume)	0, 0.2, 0.5
Load (N)	392
Sliding speed (rpm)	600
Tested at different Temperatures (°C)	40, 75, 120

In this test, wear volume that occurs on the wear scar diameter and wear scar depth can be calculated by using the following equation [ASTM D4172].

$$V = \frac{1}{2} \left(\frac{4}{3} \pi r^2 h \right) = 5.236 \times 10^{-1} \cdot d^2 \cdot h \quad (1)$$

where,

V = wear volume, mm³

d = wear scar diameter, mm

r = wear scar radius, mm

h = wear scar depth, mm

The coefficient of friction also can be found according to friction force and test load which can be calculated by using the following equation.

$$\mu = 0.00227 fL/P \quad (2)$$

where,

μ = coefficient of friction

f = friction force, N

L = length of friction lever arm, cm

P = test load, kg

3.3.4 Experimental Procedure

In this section, several processes were conducted and followed certain systematic procedure before undergoing with the experiment by using different EG concentration. First, the weighing balance is used to measure the mass of EG. Next, run the ultrasonic homogenizer. The function is to disperse the nanoparticle in the base oil via sonification technique. Then, the ultrasonic bath was utilized to clean the steel ball specimen to remove impurities, debris or dirt that can affect the results. Next, the four-ball tribometer was performed which used as wear test. The test should be run in one hour at a different temperature which are 40°C, 75°C, and 120°C at 600 rpm in speed and 392 N in load. Finally, a scanning electron microscope (SEM) is applied to check the morphology of the worn surfaces and analysis of the wear rate, coefficient of friction (COF) and wear mode under boundary lubrication. Moreover, the viscosity on friction and wear of different percentage of EG also will be investigated. To provide more detail on ultrasonic homogenizer and followed by ultrasonic bath which will be explained further on the next sub topic.

3.3.4.1 Ultrasonic Homogenizer

Ultrasonic Homogenizer functions as a tool to mix nanoparticle additives in the oil. As in this experiment, the Shell Rimula R4X SAE 15W-40 engine oil is mix together with EG by using this machine. Figure 3.5 below shows the ultrasonic homogenizer.



Figure 3.5: Ultrasonic Homogenizer

Standard of procedure:

- i. The first things need to be considered is the safety issues. Before start running the machine, the normal electrical precautions must be followed. Gloves, eye and ear protection should be wear.
 - ii. Choose the proper probe based on the total volume of the liquid.
 - iii. After that, place the probe with the spanner as a tool.
 - iv. Adjust and lift the base until the probe submerged about $\frac{1}{2}$ into the liquid container.
 - v. Next step is, tight the screw of the base.
 - vi. Switch on the machine and let the machine running about 15 minutes.
- Switch off the machine properly after use it

3.3.4.2 Ultrasonic Bath

Ultrasonic bath is used to clean specimens before and after running an experiment. Usually, the specimen will be placed in the beaker submerged with acetone. The beaker will be put in the ultrasonic bath for 10 minutes. Figure 3.6 shows the sample of ultrasonic bath.



Figure 3.6: Ultrasonic Bath

Standard of procedure:

- i. The first things needed to be considered is the safety issues. Before running the machine, the normal electrical precautions must be followed. Gloves, eye and ear protection should be worn.
- ii. Ensure the drain is empty.
- iii. Ensure the sample is submerged into acetone in a beaker.
- iv. Place or put the beaker in ultrasonic bath.
- v. Next, pour the tap water and fill the bath. Ensure that the water do not fill the pass line.
- vi. Press start button to run the bath and set time to 10 minutes.
- vii. The machine will stop after 10 minutes. Thorough cleaning is necessary after use. Drain out the tap water after use.

3.3.4.3 Ducom Four-Ball Tester

Four Ball Tester as shown in figure 3.7 can be described as a measurement device to identify the durability of lubricants by measuring the parameters such as the coefficient of friction, wear value and seizure load.

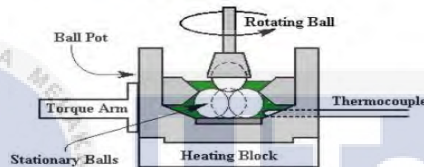


Figure 3.7: Four-Ball Tester Machine

Standard of procedure:

- i. The ball pot are cleaned with acetone and wiped with tissue paper to remove any debris, contaminants, or oil from previous run.
- ii. The ball locking ring are inserted into the ball pot and the three bottom balls are placed inside it.
- iii. The locking nut are threaded in and tightened using a torque wrench with torque rating of 40-50 N.m
- iv. The lubricant is poured until the bottom balls are almost submerged to achieve the boundary lubrication condition.
- v. The ball is inserted into the collet and secured by pressing the ball onto the collet master.

- vi. The collet is inserted into the spindle and are align by slotting in the collet into the slot.
- vii. The ball pot are transferred into the equipment and placed on the base. The ball pot needs to be align perfectly.
- viii. The hose are connected to the ball pot and the weight hanger are attached at the weight arm.
- ix. The dead weight are added to the weight hanger and the balancer are used to balance out the weight.
- x. The test parameter are keyed in into the electronic controller.
- xi. The software runs to collect real time data.
- xii. The dead weights are remove and the pot are taken out to measure the scar diameter and profile using the Spectrometer.

3.3.4.4 Weighing Balance

In the experiment, weighing balances (as shown in figure 3.8) is used to measure the mass of EG before running the experiment.

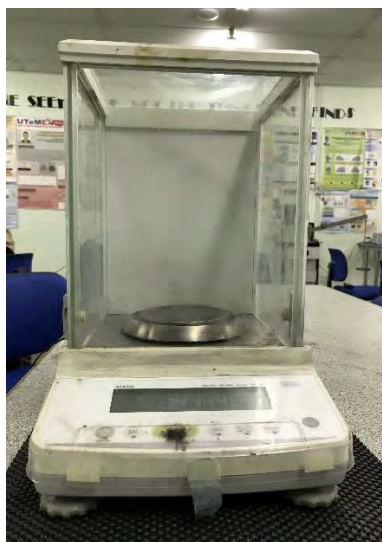


Figure 3.8: Weighing Balances

Standard of procedure:

- i. First, assure that the balance is calibrated before weighing.
- ii. In case before or during weighing supply fails, calibrate the balance once again.
- iii. The connections of the balance must be checked properly, and balance is ON.
- iv. Tare key must be press if balance showing some reading on the display. The balance should show a 0.0000 g.
- v. Open the door from one side and place the specimen, close the door and press the tare key.
- vi. Then, open the door and take out the specimen after getting the desired weight.

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3.3.4.5 Kinematic Viscosity Apparatus

In the experiment, the kinematic viscosity apparatus is used to measure the viscosity of 0.2% and 0.5% mixing oil after running the experiment. Figure 3.9 shows the example of kinematic viscosity apparatus.



Figure 3.9: Kinematic Viscosity Apparatus

Standard of procedure:

- i. First, turn on the switch, power, and heating. Stir before performing kinematic viscosity.
- ii. Set up the temperature on the controller until 40 °C.
- iii. Choose the size 1.55 mm of tube according to viscosity range.
- iv. Put oil inside the tube until it reaches the point marked between G and H.
- v. Lock the tube to the holder (clip) and put it in the viscometer.
- vi. Make sure the tube is vertical by using the alignment device.
- vii. Wait until the thermometer and controller shows constant temperature.
- viii. Install the pipette (sucker ball) on top of the N tube and start suck the oil above mark E.
- ix. Release the cover from tube N and start timing required for the oil pass from mark E to mark F.
- x. If the flow time is less than 200s, select a smaller capillary tube size.

- xi. Take out the tube and clean thoroughly by using the appropriate solution and dry the tube by flowing dry air until the last trace of liquid removed.

3.3.5 Surface Analysis

The friction force on the contacting sliding surface and the temperature of the lubricant in the oil bath are continuously monitored and recorded using a PC data acquisition system. After the wear test completed, various characterisation of the specimen will be performed. The wear mode of the specimen will be measured using an optical microscope. A scanning electron microscope (SEM) is applied to check the morphology of the worn surfaces and to achieve the wear scar diameter. The chemical films are analyzed using energy dispersive X-ray (EDX) analysis which provides a semi-qualitative analysis of the tribofilm composition on the wear surface.



CHAPTER 4

RESULTS AND ANALYSIS

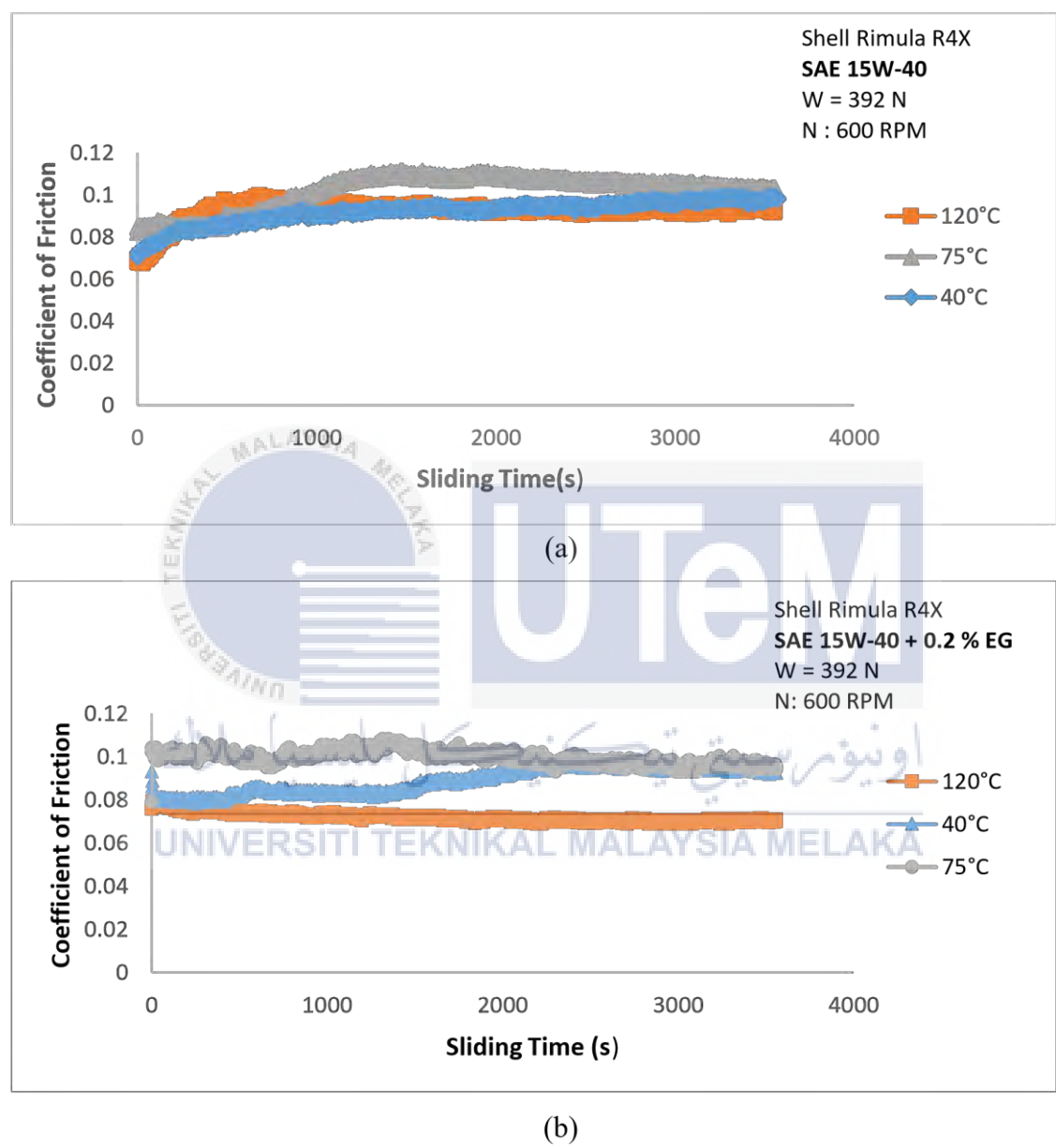
4.1 Coefficient of Friction

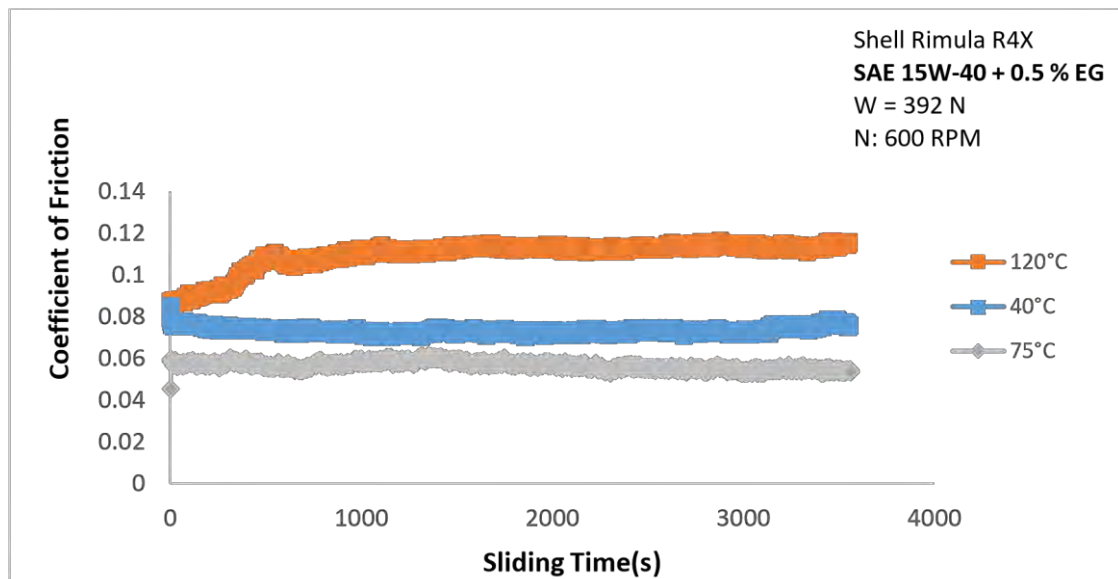
Figure 4.1(a), (b) and (c) shows the result of coefficient of friction (COF) against sliding time L [sec] at different temperatures T [°C] for different percentage of expanded graphite (EG) in Shell Rimula R4X SAE 15W-40. From the graph, the maximum peak of COF for Shell Rimula R4X SAE 15W-40 oil is 0.12, which is higher than 0.2% and 0.5%. Expanded Graphite (EG) has a value of 0.1170 and 0.1157. The three base oil was run with the same load and speed of 392 N and 600 rpm respectively. This meant that the engine oil with 0.5% EG produced a small value of COF compared to others. This satisfies the tribological properties in order to reduce friction and wear.

Figure 4.1(b) shows the results acquired by testing the Shell Rimula R4X SAE 15W40 + 0.2 % EG. The three temperatures displayed in the graph shows similar results. At 120°C, it shows that there is a lower friction coefficient compared to other temperatures. At 40°C the figure shows a friction coefficient that increases slightly during 1500s. After that, the coefficient stabilizes to a constant value.

For Shell Rimula R4X SAE 15W-40 + 0.5 % EG as shown in Figure 4.1(c), the friction torque at 120°C was increased rapidly in the beginning of the experiment and then slowed to a steady state at the end of the experiment. Temperatures 40°C and 75°C showed

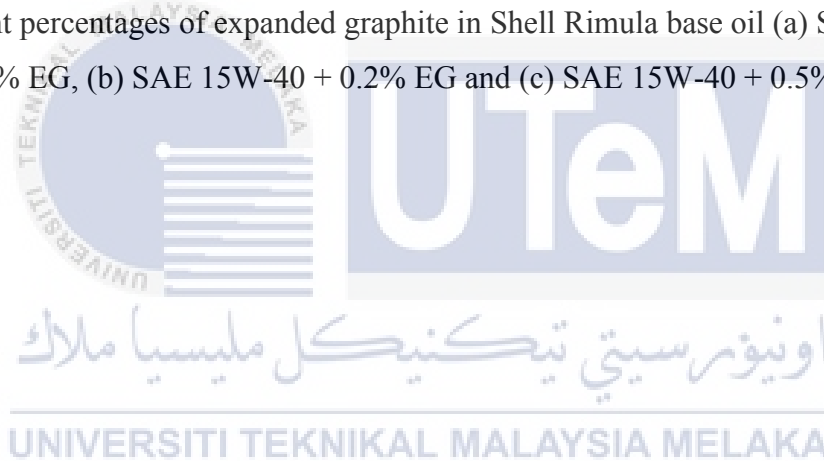
a steady condition until the end of the experiment. The steady condition of friction coefficient represents that the lubricant layer between steel balls was stable and no critical damage occurred in the lubrication film.





(c)

Figure 4.1 Coefficient of friction (COF) against sliding time (s) at 40°C, 75°C and 120°C for different percentages of expanded graphite in Shell Rimula base oil (a) SAE 15W-40 + 0% EG, (b) SAE 15W-40 + 0.2% EG and (c) SAE 15W-40 + 0.5% EG.



4.2 Coefficient of Friction at stable region

Figure 4.2 shows the graph of Coefficient of Friction (COF) against different temperatures at a stable region. Stable condition of the friction torque shows that the lubrication layer between ball-bearings was stable and no severe breakdown of lubricant film occurred. When compared three engine oils against temperatures of 40°C, 75°C and 120°C, there is a stark contrast between the results. Based on 40°C graph, the maximum peak is at 0.2% EG and the lowest peak is 0.5%.

At 75°C, the EG percentage has a remarkable difference between 0% and 0.5%. This meant that engine oil with 0.5% EG has the best coefficient of friction at a stable engine running condition. Hence, it reached a stable region faster than the engine oil with 0.2% EG and non-additive are still in the running in condition which reduce effect of weight and volume loss of the steel ball.

At higher temperature of 120°C, the oil with 0.2% EG has a lower coefficient of friction compared to 0.5%. This means that the engine oil with 0.5% EG is suitable for the application at normal engine running temperature in order to minimize friction.

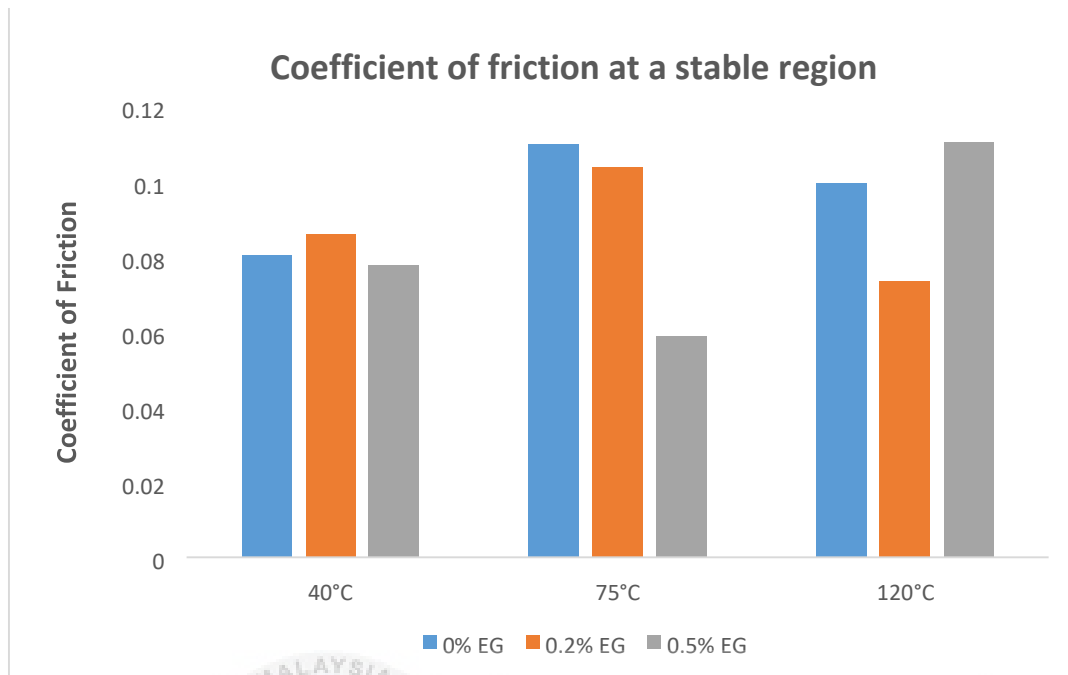
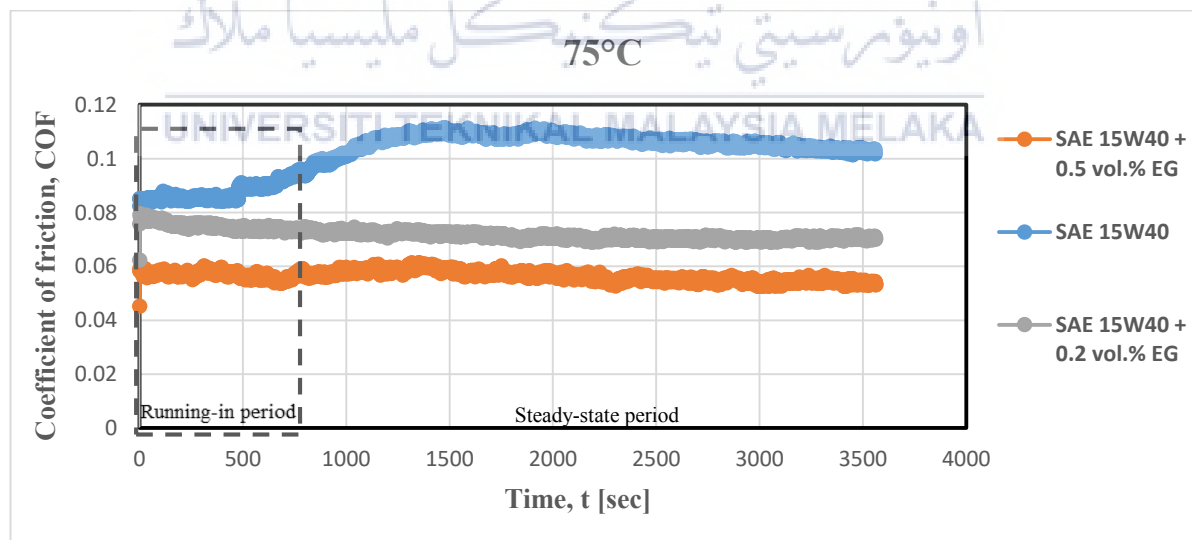
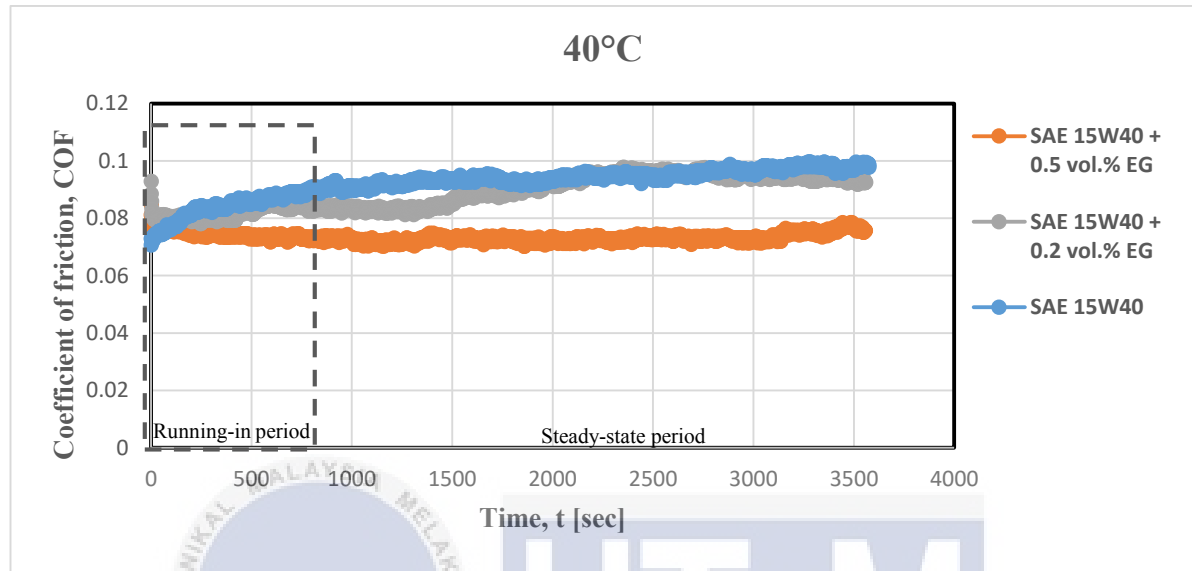


Figure 4.2 : Graph of Coefficient of friction against different temperature at Stable Region

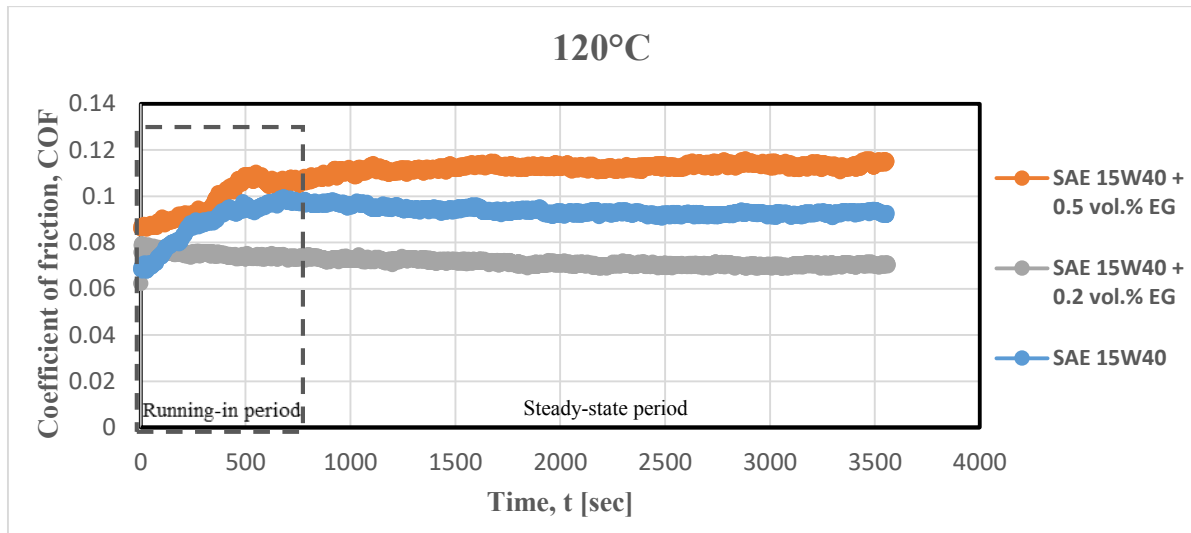
4.3 Coefficient of Friction at Running in Region

Running-in is an engine condition where the engine starts to move and is subjected to increasing heat. The heat then stabilizes itself depending on the oil. This clears up or readjust the engine components and generally, throughout this period, the oil consumption might be higher. The characteristics of frictional running-in include: the overall trend in friction force with time, the duration of characteristic features in the friction/time curve, and the instantaneous level of frictional fluctuations superimposed on the general trend. Changes in friction and wear that occur during running-in are more than a consequence of surface roughness alterations alone. Depending on the tribosystem, they can also include changes in surface composition, microstructure, and third-body distribution. Figure 4.3 (a) shows the graph of Coefficient of Friction against Sliding Time with a 40°C Temperature at Different Percentages of EG. From the graph, the highest peak of coefficient of friction is at 0% EG which is greater than 0.2% and 0.5% EG. This means that the engine oil with 0% has engine loss and high amounts of friction that can lead to a few mechanical problems when compared

to others with additives in it. Overall, engine oils with 0.2% and 0.5% EG have better CoF and improves running-in. It is definitely an effective oil when it comes to reduction of friction.



(b)



(c)

Figure 4.3: Coefficient of friction against sliding time with temperature for different percentage of EG at (a) 40°C (b) 75°C and (c) 120°C.

4.4 Wear Scar Diameter

At the end of 1 hour steady state tests, the worn surface of the steel ball has been measured by using winRoof optical microscopes to determine the wear scar diameter (WSD). Figure 4.4 shows the wear scar diameter morphology of steel ball. Smaller wear scar diameters have been observed at the end of the tests that has been carried out with lubricants sampled with different EG contents. These results ensure that in boundary lubrication condition the presence of EG mix with engine oil encourages chemical reactions in the surface and as a result reduces friction and wear. The WSD values are reported in Table 4.1.

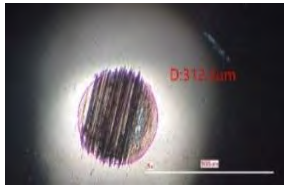
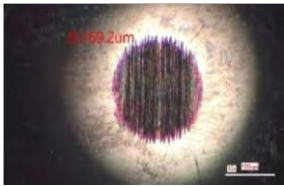
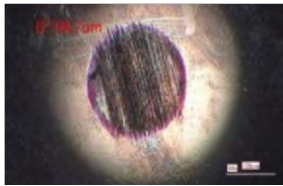
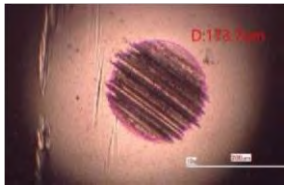
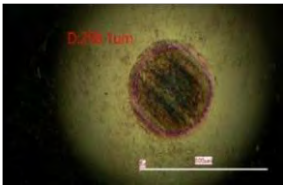
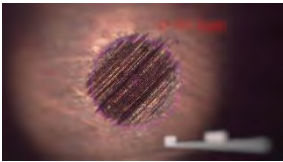


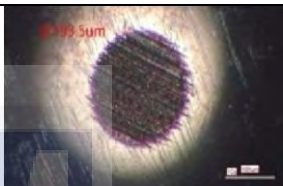
	40°C	75°C	120°C
0% EG			
0.2% EG			
0.5% EG			

Figure 4.4: Images of steel ball wear scar

Table 4.1: Wear Scar Diameter in mm

	0% EG	0.2% EG	0.5% EG
WSD at 40°C [mm]	0.3122	0.1736	0.3004
WSD at 75°C [mm]	0.1692	0.2980	0.1858
WSD at 120°C[mm]	0.1946	0.53	0.1935

From the experimental data, wear scar diameter is observed in order to find the wear volume. The wear volume against different temperatures will be compared among the three oils. This is done to determine the lowest wear volume between additive and non-additive oils. From the graph pattern in figure 4.5, the results at 75°C showed a reduction of wear scar diameter

that has a concentration of 0% additive. Moreover, 0.2% EG has the biggest wear scar diameter compared to the others.

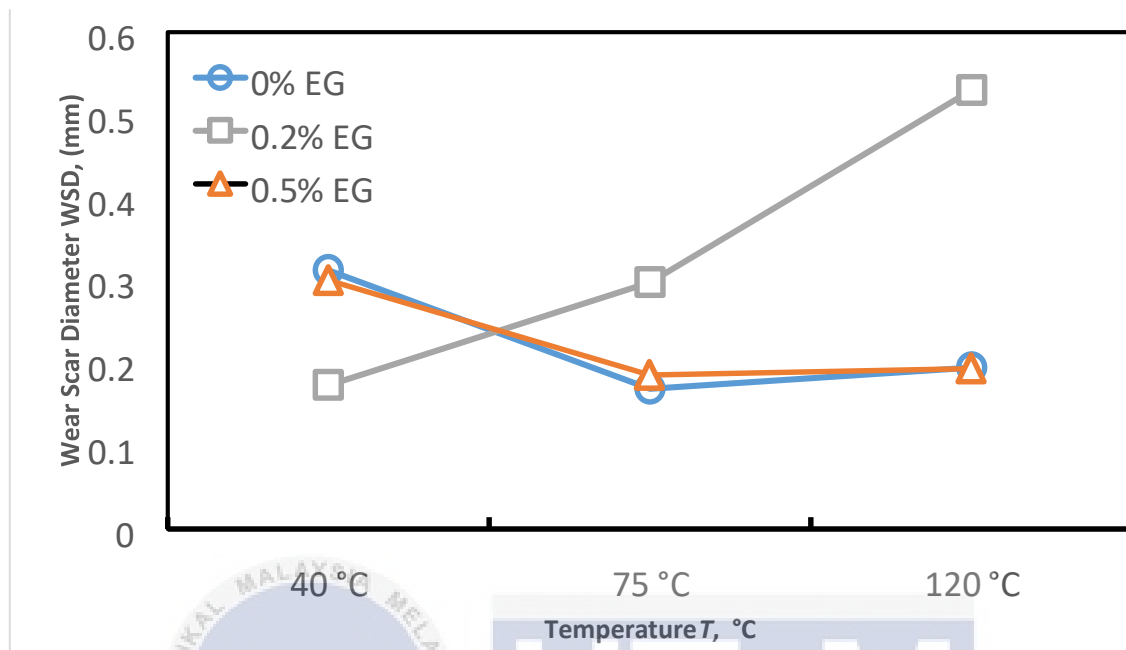


Figure 4.5: Wear Scar Diameter of steel ball at different temperature and percentage of expanded graphite in base engine oil

4.5 Wear volume k against Different Temperature, °C

Figure 4.6 shows the effect of lubricating condition on wear volume of 0%, 0.2% and 0.5% EG. From the figure, 0.5% EG has the minimum value of wear volume. This was observed in oil lubrication condition and also in oil with EG lubrication. For 0% and 0.5%, both of these show the smallest wear volume in base engine oil when compared to 0.2% EG. Therefore, 0.5% contains better wear properties in additive and non-additive oil. The figure also shows 0.5% EG maintains an almost constant wear volume as the temperature increases. The wear volume against different temperatures for 0% EG, 0.2% EG and 0.5% EG values are reported in Table 4.2.

Table 4.2: Wear Volume for 0% EG, 0.2% EG and 0.5% EG

Temperature, °C	Wear Volume, k for Shell Rimula R4X SAE 15W-40- $\frac{mm^3}{Nm}$	Wear Volume, k for Shell Rimula R4X SAE 15W-40 + 0.2% EG, $\frac{mm^3}{Nm}$	Wear Volume, k for Shell Rimula R4X SAE 15W-40 + 0.5% EG, $\frac{mm^3}{Nm}$
40°C	7.34	7.02	6.29
75°C	6.34	6.1	9.21
120°C	1.11	0.00061	1.08

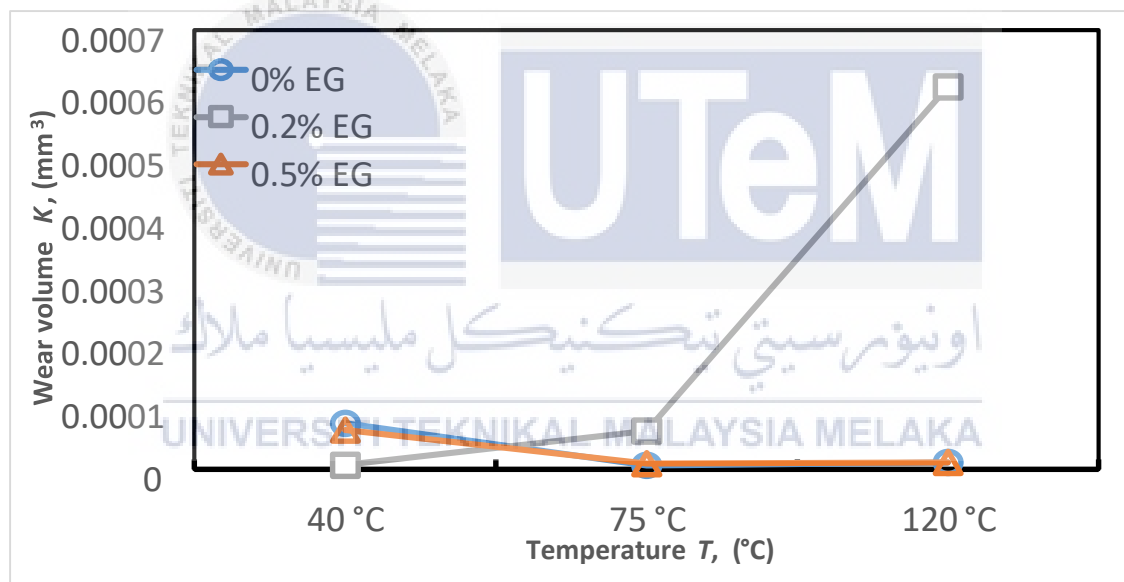


Figure 4.6: Graph of Wear Volume (mm³) against Temperature (°C)

4.6 Wear scar observation on 0% and 0.5% EG at temperature 75°C

Due to the EG additive being significantly easier to absorb upon the rubbing surfaces in mineral base oil, the base oil with the additive was more compatible when compared to the oil without any additives added. Great absorption of additive contained in mixed oil on the rubbing surface exhibits good compatibility between the additive and the base oil[22].

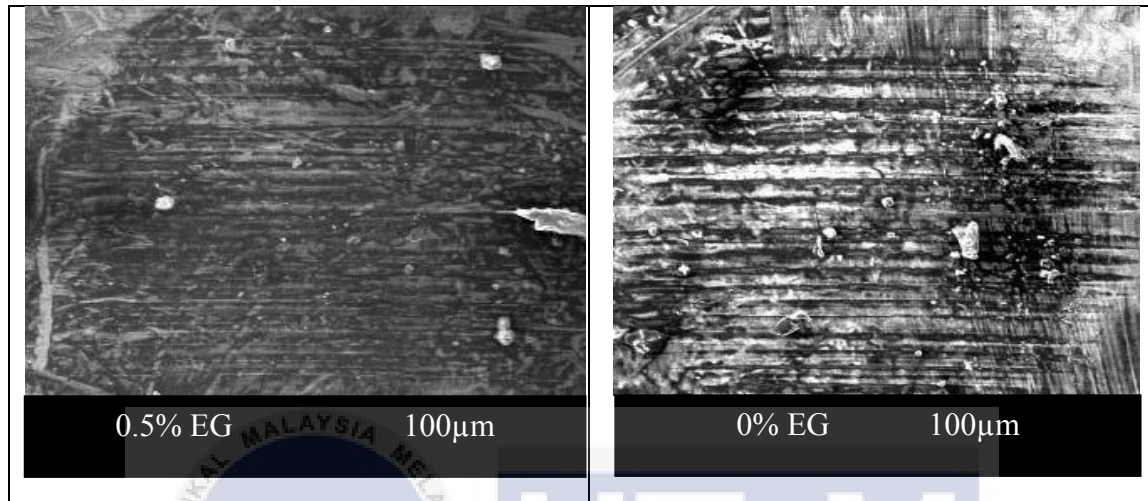
Therefore, the EG additive forms a protective film easier on the rubbing surfaces when it is in mineral base oil. The greater absorption of the additive is associated with a more extensive chemical reaction upon rubbing of the steel balls. As well as a shorter replenishment time of the protective film. SEM was employed to qualitatively determine the tribofilm composition and thus predict this phenomenon.

Figure 4.7 (a) shows SEM images of the wear scar of the ball specimen for experimental condition with load 392N. The worn surface on the ball-bearing are lubricated with both 0% and 0.5% EG. It is tested at 0.5% volume of EG which is proven to be the most optimal composition of additive compared with the original one which is 0% additive. Then, the test were performed at the most optimal temperature in the Coefficient of Friction which is 75°C. They show different wear patterns with parallel grooves. From the figure, 0.5% EG has shallow scratches when compared to 0% EG which has more deep grooves. Due stiff particles; like wear debris of the oxide layer, and ragged adhesion, this results in a deeper, significant groove pattern[23].

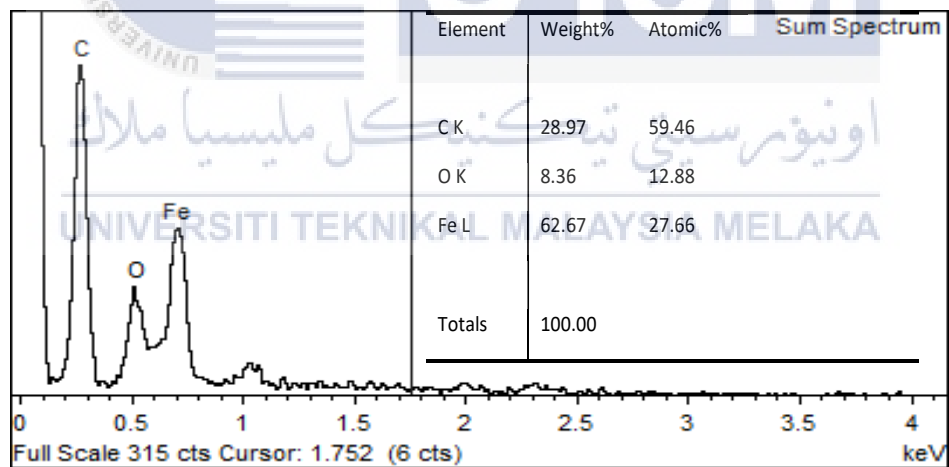
Based on a test using 0% EG with load 392 N at an operating temperature of 75°C, it observed that Fe elements have a higher weight percentage when compared to C and O₂ elements on the wear scar diameter as shown in figure 4.7 (b). Fe elements are at 62.67% whereas C elements are at 28.97% and O₂ elements are at 8.36% respectively. The results imply that the rubbing effect under 392 N load cannot promote a chemical reaction, in this case; forming a protective film, due to lack of C elements contained in the additive.

However, based on a test of 0.5% EG with 392N load at 75°C within figure 4.7 (c), contains a higher percentage of C and Fe elements when compared with only a small percentage of O₂ and Zn elements. The chemical reaction of the additive is triggered, thus

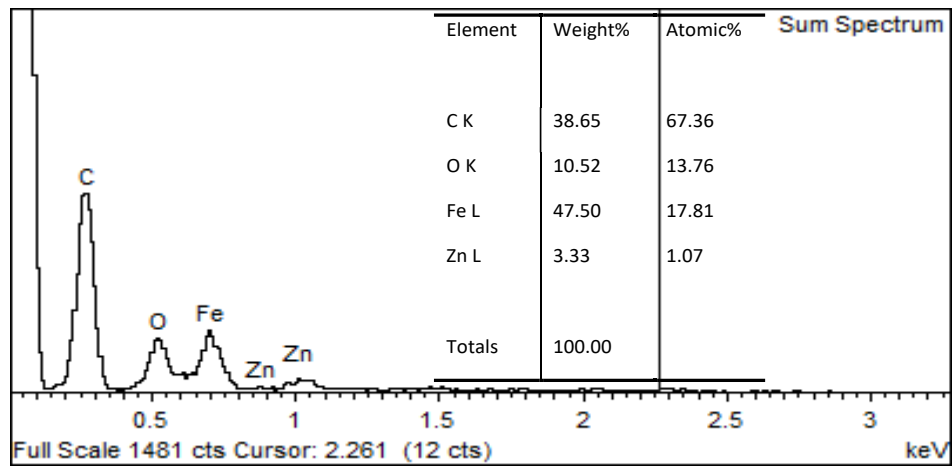
causing the formation of Carbon to be accompanied with tribochemical wear behavior. Lastly, the reaction rate of additive in chemical film formed by tribochemical reaction will dominate the wear behavior.



(a)



(b)



(c)

Figure 4.7 (a): SEM images of the wear scars at an applied load of 392N on the ball specimen
 (b) SEM spectra at spots specified in 392 N load in Mineral base oil with 0% EG additives
 (c) SEM spectra at spots specified in 392 N load in Mineral base oil with 0.5% EG additives



CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

To conclude, based on the results obtained from the experiment, mineral engine oil with 0.5% Expanded Graphite (EG) nanoparticles at 75°C has been recorded as the lowest coefficient of friction and wear volume when compared to the controlled mineral engine oil and mineral engine oil with 0.2% EG at temperature 40°C and 120°C . All engine oil has been tested with the same parameter: same load and speed at different temperatures. Engine oil with EG show the positive results and fulfil the aim of reducing friction and wear. Other than that, the volume of EG which is 0.5% has proven to be the most optimal composition of additive added into mineral oil. The effects can be seen in the wear volume graph which shows a significant difference when compared to other percentages of EG. Then, the most optimal temperature in the Coefficient of Friction is at 75°C. This can be seen in graph of COF against sliding time with temperature which indicates a major difference between 40°C and 120°C. Besides that, engine oil with 0.5% EG is the most stable in COF when compared to 0% and 0.5%. From the overall study on the behavior of tribological properties, EG nanoparticles is the most effective and suitable additive in engine oil since it reduces friction and wear at the most optimum temperature. Hence, it can makes a car's engine perform efficiently since there is low friction and wear in the engine's component.

5.2 Recommendation

Several recommendations for future works on this project are listed below:

- i) A study on the effects of mineral oil mixed with EG additives which has been left sitting covered and uncovered for a month. This is to understand any occurrence of separation between the oil and the additive.
- ii) Analyse SEM and EDX on every sample which is 0,0.2 and 0.5%
- iii) Add antiwear properties such as HbN additives



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