TRIBOLOGICAL ANALYSIS OF STEEL AGAINST STEEL CONTACT UNDER EXTREME BY USING DIFFERENT TYPES OF ANTI-WEAR AND FRICTION ADDITIVES

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DECLARATION

I declare that this project report entitled "Tribological Analysis of Steel Against Steel Contact Under Extreme Pressure By Using Different Types of Anti-Wear and Friction Additives" is the result of my own work except as cited in the references

Signature : ....

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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 (with Honours)

Signature : ………………………………..

Name of Supervisor : DR MOHD RODY BIN MOHAMAD ZIN

Date : …………………………………

DEDICATION

To my beloved mother, Fatimah Bt Ariffin and loving father, Abdull Jalil Bin Ahmad, for their endless support and assurance through all of my journey

ABSTRACT

This study aims to investigate the extreme pressure properties of Alumina (Al_2O_3) nanoparticles as an additive in SAE 15W-40 Diesel Engine Oil to improve the extreme pressure properties of the base oil. Conventional base oil additive failed to provide protective lubrication layer under extreme pressure (EP) and prevent engine seizure. Base oil without nanoparticle also have lower load carrying capability and have high Coefficient of Friction (CoF) and other friction reduction method such as Diamond Like Carbon Coating (DLC) are much more complex and costly. Therefore, low cost and environmentally nanoparticle which is Alumina (Al_2O_3) and Hexagonal Boron Nitride (hBN) are chosen to be used as the extreme pressure additive in diesel engine oil to improve the load carrying capability and reduce the CoF of the base oil itself. The nano-oil are prepared by dispersing hBN and Al_2O_3 nanoparticle with an optimal concentration of 0.5 vol % into the diesel engine oil using sonication method via ultrasonic homogenizer. The tribological test were performed using 4-Ball Tester in accordance of ASTM D 2596 and D 2783 standards. Based on the experimental results, it is found that Al_2O_3 has the potential to improve the extreme pressure properties of the base oil where it reduces the severe adhesive wear and increase the load carrying capability almost twice the load carrying capability of the base oil from 618N to 1236N. Al_2O_3 nanoparticle are also able to reduce the Coefficient of Friction (CoF) up to 48% .These finding are then verified using Scanning Electron Microscope (SEM) and Electron Dispersive X-Ray (EDX) analysis where it is found that Al element are present on the worn surface. Therefore, Al_2O_3 nanoparticles are believed to play three lubricating mechanism which is ball bearing effect, mending effect and tribofilm formation that led to the improvement of the extreme pressure of the base oil. Both nanoparticle plays the same lubricating mechanism however, they display different extreme pressure properties where Al_2O_3 nanoparticles has better extreme pressure properties than hBN nanoparticles as the point of the last non-seizure of Al_2O_3 are higher at 1236N compared to 786N of the hBN nanoparticles. Therefore, the usage of nanoparticle as an extreme pressure additive has significantly improved the load carrying capability and lower coefficient of friction which is significant for the application in modern internal combustion engine.

ABSTRAK

Kajian ini dijalankan untuk menyiasat sifat tekanan ekstrem nanopartikel Alumina (Al2O3) sebagai aditif dalam minyak pelincir enjin diesel SAE 15W-40 untuk memperbaiki sifat tekanan ekstrem minyak asas. Campuran minyak enjin konvensional gagal memberikan perlindungan di bawah tekanan melampau dan mencegah kerosakkan dan kegagalan enjin. Minyak enjin tanpa nanopartikel juga mempunyai keupayaan membawa beban yang lebih rendah dan mempunyai Pekali Geseran yang tinggi (CoF) dan kaedah pengurangan geseran lain seperti salutan karbon seperti berlian (DLC) adalah lebih kompleks dan mahal. Oleh itu,nanopartikel kos rendah dan mesra alam sekitar seperti Alumina (Al_2O_3) dan Boron Nitrida Heksagonal (hBN) dipilih untuk digunakan sebagai aditif tekanan ekstrem dalam minyak enjin diesel untuk meningkatkan keupayaan membawa beban dan mengurangkan pekali geseran minyak asas itu sendiri. Minyak nano disediakan dengan menyebarkan nanopartikel hBN dan Al_2O_3 dengan kepekatan optimum 0.5 vol% ke dalam minyak enjin diesel menggunakan kaedah sonikasi melalui homogenizer ultrasonik. Ujian tribologi dilakukan menggunakan Penguji 4-Bola mengikut piawaian ASTM D 2596 dan D 2783. Berdasarkan keputusan eksperimen,nanopartikel Al_2O_3 didapati mempunyai potensi untuk meningkatkan sifat tekanan melampau minyak enjin di mana ia mengurangkan kehausan yang teruk dan meningkatkan keupayaan membawa beban hampir dua kali ganda keupayaan membawa beban minyak enjin dari 618N ke 1236N. Nanopartikel Al_2O_3 juga mampu mengurangkan pekali geseran sehingga 48%. Penemuan ini kemudiannya disahkan menggunakan analisis Mikroskop Pengimbas Elektron (SEM) dan X-Ray Penyeberan Elektron (EDX) di mana ia didapati bahawa elemen Al hadir pada permukaan geseran. Oleh itu, nanopartikel Al_2O_3 dipercayai memainkan tiga mekanisme pelinciran yang merupakan kesan bebola galas, kesan pembaikan dan pembentukan tribofilm yang membawa kepada peningkatan sifat tekanan ekstrem.Kedua-dua nanopartikel memainkan mekanisma pelincir yang sama namun, mereka memaparkan ciri-ciri tekanan ekstrem yang berlainan di mana nanopartikel Al_2O_3 mempunyai sifat tekanan ekstrem yang lebih baik daripada nanopartikel hBN di mana titik terakhir tanpa kerosakan Al_2O_3 yang lebih tinggi pada 1236N berbanding dengan 786N dari nanopartikel hBN. Oleh itu, penggunaan nanopartikel sebagai aditif tekanan ekstrem meningkatkan keupayaan membawa beban dan pekali geseran yang lebih rendah di mana ia penting bagi aplikasi enjin pembakaran dalaman moden.

ACKNOWLEDGEMENT

I would like to dedicate this study finding and discovery for the future reference of new researcher in Tribology field and Faculty of Mechanical Engineering.

For the unconditional support, advices, and guidance given by Dr Mohd Rody Bin Mohamad Zin, I highly appreciate all he have put in and could not thank him enough. For the motivating comment and review given by my panellist, P.M Dr Fadzli Bin Abdollah and Dr Kamal Bin Mat Yamin which allows me to improve the quality of this study, I dedicated a sense of gratitude and thankfulness. To the technical knowledge and assistance offered by Ms Ayuma and Mdm Hidayah which enabled my experiment and testing to take place smoothly and accordingly, I sincerely thank them both.

To my friend and classmates for their opinion and sharing of information. To all the person that have help directly or indirectly, thank you for your contribution for making this study possible and achievable.

Most of importantly, to our great Almighty God, the owner of knowledge and wisdom for allowing everything to takes places and discovered.

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LIST OF ABBEREVATIONS

Al_2O_3	-	Aluminum Oxide
ASTM	-	American Society for Testing and Materials
API	-	American Petroleum Institute
BSFC	-	Brake Specific Fuel Consumption
COF	-	Coefficient of Friction
DLC	-	Diamond Like Coating
EDX	-	Electron Dispersive X-Ray
hBN	-	hexagonal Boron Nitride
HRC	-	Rockwell C Hardness
ICE	-	Internal Combustion Engine
LWI	-	Load Wear Index
PPE	-	Personal Protective Equipment
SAE	-	Society of Automotive Engineer
SDS	-	Sodium Dodecyl Sulphate
SEM	-	Scanning Electron Microscopy
TAN	-	Total Acid Number
TBN	-	Total Base Number
VI	-	Viscosity Index
WSD	-	Wear Scar Diameter
ZDDP	-	Zinc Dialkyldithiophosphates

LIST OF SYMBOL

μ	-	coefficient of frictiom
f	-	friction force, N
L	-	Length of friction lever arm
P	-	test load , kg
A	-	sum of the corrected load determined for the ten applied loads immediately before weld point
L	-	applied load total load multiplied by lever arm ratio
Dh	-	Hertz scar diameter, mm
X	-	average scar diameter
Dh	-	Hertz diameter of the contact area,
P	-	the static applied load

CHAPTER 1

INTRODUCTION

1.1 Background

Tribology can be defined as the study of two interacting surface which mainly focussing on three principal which is wear, friction and lubrication [1]. Friction and wear are generated when two different surfaces are in direct contact which causes rotational or sliding resistances due to different surface Coefficient of Friction (CoF). The increase in demand of reducing friction and wear has introduce new material to tribology study which is nanomaterial. Nanomaterial have attract numerous industry and researchers to apply nanoparticle as new form of additives especially in lubrication technology based on Dei et al. [2]. The dispersion of nanoparticle into lubricant forms a new type of lubricant known as nanolubricant. Nanolubricant are made of three components which is fully formulated lubricants or base oil, with colloidal solid particles also known as solid lubricant and surfactant [3]. The solid lubricant or nanoparticles serves a special function which is anti-wear or extreme pressure (EP) additives and the surfactant prevents agglomeration of the nanoparticles [4]

As Tribology field evolves, there have been various exciting and successful finding which shows that nanoparticles serve significant improvement in lubricating properties. One of the previous finding states that the addition of spark plasma sintered-Al_2O_3 composites containing barite-type structure sulphates [5] and chromium oxide (CrO) nanoparticle in lubricating oil [6] steadily improve the extreme pressure properties and lower down the CoF that extends the bearing life and susceptible to seizure. The addition of 10nm Nickel nanoparticle in oil with varying concentration of 0.2-0.5% had increased the non-seizure load by 67 %, reduction in wear scar diameter (WSD) by 22% and friction coefficient reduction of 26% compared to the base oil as discovered by Qiu et al. [7]. Therefore, this study is inspired from other finding to analyse the tribological performance of Al_2O_3 and hBN nanoparticles under extreme pressure. Both of this nanoparticles are environmental friendly and low cost making it interesting to be applied into a real application. In this study, 70nm of both nanoparticle are dispersed in SAE 15W-40 mineral diesel engine oil via sonication technique and tribologically tested using 4-Ball Tester. The testing procedure adhere to ASTM D 2596 and D 2783 standards for extreme pressure test under boundary lubrication regime. The improvement in terms of extreme pressure and load carrying capability are then investigated further via SEM and EDX analysis. After such analysis are done, the best nanoparticle to be used as engine oil additive will be chosen which is Al_2O_3 as it shows improvement in extreme pressure properties and reduction in CoF and non-seizure load.

1.2 Problem Statement

With the advancement of material and engine component, lubrication plays a major role in allowing an engine to achieve good Brake Specific Fuel Consumption (BSFC), excellent thermal efficiency, as well as extending the engine life span. Engine oil are demanded to provide protection of metal on metal under EP to reduce wear and prevent catastrophic cases such as engine seizure from occurring where conventional engine oil without nano-additive fail to do so.

The usage of nano-additive are known to be able to improve the important lubrication properties such as Viscosity Index (VI), Total Base Number (TBN), Oxidation resistance and high temperature performance. As the key properties of base oil are improved, the ability of the lubricant to reduce wear rate, CoF, WSD and load carrying capability also improve significantly. Excellent load carrying capability enable seizure to occur at higher load which allows engine to perform exceptionally better even under harsh environment. Furthermore, most research are done only to study the tribological performance of Graphene based additive such as hexagonal Boron Nitride (hBN) or Graphene Oxide rather than Oxide based additive such as Aluminium Oxide (Al_2O_3) under EP.

Therefore, nano-additive is required to be formulated with conventional base oil to improve the tribological performance of the base oil itself. In addition, there is also a need to compare the difference in tribological performance of hBN and Al_2O_3 which both are environmentally friendly and relatively low cost compared to other friction reduction method such as diamond like carbon coating (DLC) which are not available to the normal consumer and any outgoing engine

1.3 Objective

1. To investigate the Extreme Pressure properties of Al_2O_3 nanoparticles as an additive in diesel engine oil
2. To identify the best nanoparticles (alumina and hexagonal boron nitride) in diesel oil engine

1.4 Scope of Project

The scopes of this project are:

1. Only results of WSD against load, SEM and EDX analysis for Al_2O_3 are presented in this report. The results of hBN and base oil against load are obtained from another study conducted by an associate professor from UTeM in his journal titled as "*Effect of hexagonal boron nitride nanoparticles as an additive on the extreme pressure properties of engine oil*"
2. This study uses Al_2O_3 as the nanoparticle to be dispersed in Shell Rimula R4X SAE 15W-40 Mineral Diesel Engine Oil which will then referred as base oil
3. The sample are made by dispersing 0.5 vol % of Al_2O_3 in 150 ml of base oil via sonication using Sartorius Stedim Labsonic P ultrasonic homogenizer for 80 minutes.
4. Tribological test are done using Ducom 4 ball tester under ASTM D 2596 and D 2783 standards
5. The quantitative analysis are done using 3D Non-contact Profilometer to obtain the WSD and wear profile surface.
6. The results obtained are used to compare the tribological performance of hBN and Al_2O_3 nanoparticle in terms of Wear Scar Diameter against Load.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Friction between two rubbing parts is the greatest limitation of ICE both Diesel and Gasoline engine. Lowering friction has become the ultimate goal to enhance both mechanical and fuel efficiency to further extract more output power per fuel mixture burned. In the early days of the ICE being engineered, they are very inefficient and lubrication technology is somehow limited with short drain interval and creates pollutant to the environment. Modern engine could achieve 30-35% efficiency for the fuel combusted where the rest of them are loss in various way. Ahmed Ali et al. found that the major power loss are highest in the engine itself especially in the reciprocating component which is piston ring and the cylinder bore assembly. This is where lubricant plays it role especially nanolubricant with their small molecular size enabling them to squeeze through tight space providing extra protection.

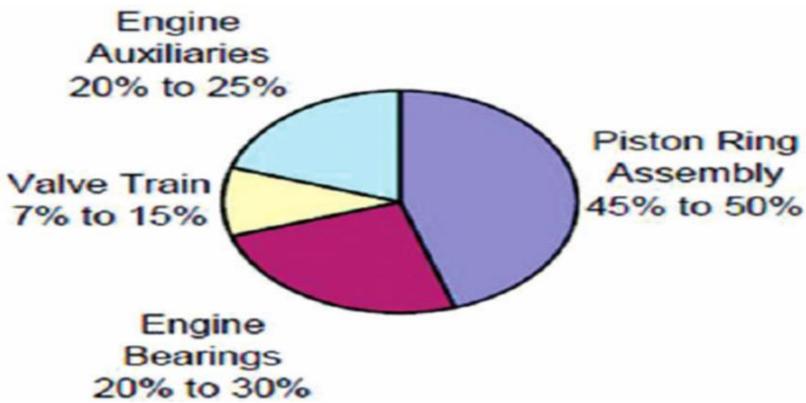

Figure 2.1 : Engine power loss for ICE

[Source : Ahmed Ali et al. [8]]

Lubrication is the key enabling factor to allow an engine to operate in a harsh environment such as extreme temperature, thermal stress and pressure generated from moving and rubbing metal. They work by separating the moving and rubbing metal by forming a protective layer of tribofilm to minimize direct contact and in turn, reduce heat and wear rate. When mentioning about lubrication, we need to observe a Stribeck curve which illustrate the lubrication regime of a lubricant. In Figure 2.2 below, there are three lubrication regime that we need to understand in which the only difference is the thickness of the lubricating film. In boundary lubrication regime, the friction coefficient is the highest since this is where most direct contact of metal occurs. A combination of high load at low rpm worsening the wear and friction which commonly happened during initial start-up of the engine as the oil pump takes some time to feed pressurized oil from the oil sump to all critical parts of engine. Critical engine parts such as crankshaft journal bearing, valve train, and cylinder bore will be the most affected making EP additive the most important aspect to be added in an engine oil. As the pressurized engine oil are supplied by the oil pump, moving components such as journal bearing are floated and the direct contact of metal are now inhibited making the friction coefficient significantly lower as the lubrication regime has shifted from boundary regime to hydrodynamic and elasto-hydrodynamic. Nanolubricant has a significant properties compared to conventional lubricant where they have high viscosity index making them better at maintaining stable film thin film. This enable the critical engine component to work under extreme pressure effectively and the overall engine efficiency can be increased significantly [1].

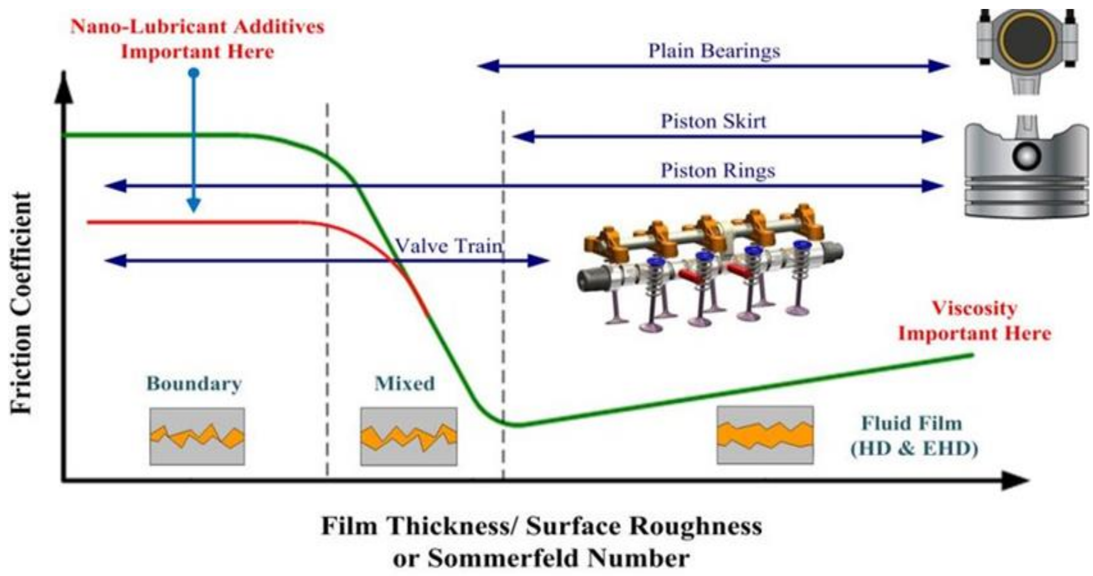

Figure 2.2 : Stribeck curve demonstrating the three different lubrication regime and the nanoparticle role

[Source : Xianjun et al. [9]]

2.2 Nano-Additive as lubricant enhancer

Before nano-additive was discovered, the automotive industry have been relying on macro and microscopic additive to enhance the performance of the base oil back in 1999. Macro and micro additives are found to be inadequate where it cannot reduce friction efficiently since the particle are large in size making them unable to seep through critical area of an engine. One of the most widely used additives is zinc dialkyldithiophosphates (ZDDP) to provide protection against wear where it is found since 1940. ZDDP possess antioxidant properties making it the best in both worlds in terms of reducing wear and as antioxidants. However, ZDDP contain harmful element such as ash, sulphur and phosphorus making it unfriendly to the environment forcing the lubrication industry to seek environmental friendly oil additives with lesser harmful element than ZDDP. This is where nano-additive are discovered and make way into lubrication technology where nano-sized particle with the grain size of 10^9

of a meter are added into the lubricant to control adhesion, friction, stiction and wear at a scale where these nano-sized particles control the outcome [10]. There are five potential advantages of using nano-additives such as (1) insolubility in non-polar base oils, (2) low reactivity with other additives in typical lubricants ,(3) high possibility of film formation on many types of surface (4) higher durability ,(5) high temperature tolerant as mentioned by Spikes et al. [11] . There are a number of effective method to reduce engine friction such as DLC. However, DLC is not readily available and still in research and development phase although few giant manufacturer such as Nissan Motor Company already implement this technologies. As reported by Kano et al., the significance and mechanism of friction reduction mechanism is difficult to studies since the film of the DLC are 1 μm in size and the interactions of DLC with lubricant additives such as ZDDP is not yet establish [12] . In addition, DLC also only been managed to be applied at limited component such as valve lifter, piston rings and valve bucket. Therefore, the usage of nano-additives particle is still relevant considering DLC is not widely available to the current and the previous engine in the market.

2.3 Optimum concentration of nano-additive

When dispersing nano-additive, the concentration of the additive is crucial to ensure optimum tribological performance. Too low in concentration would be insufficient and the friction reducing mechanism would be insignificant. Too high in concentration would be excessive and it will effects other additive such detergent and dispersant additive performance. From previous studies, it is reported that the optimal concentration of a nano-oil ranges from 0.1-2.0 % to which proven to provide reduction in CoF and wear rate as reported by other researcher. Mouquan et al. reported that the best concentration of 50 -150nm ALF_3 is 1 wt % when it is added into 100 SN base oil where it exhibits excellent friction reducing and anti-wear properties [10].

Ravindra et al. uses silicon oxides(SiO_2) and Nickel carbon coated when it is added into SAE 10W-30 base oil and found that the best concentration was 1 wt % and the friction reduction are reduced by 8%-35% meanwhile the rate of wear are reduced by 4.6%-23% [13]. Nguyen et al. discovered that the optimal concentration of aluminium nanoparticle is 0.67 wt % when it is added into glycerol lubricants with 2 wt % of sodium dodecyl sulphate (SDS) as dispersive medium and 10 wt % of deionized water content of glycerol. The finding is that able to reduce surface roughness remarkably [14]. For carbon based nanoparticles, it is observed that the concentration used is lower compared to metal oxide particles. For real world application, high concentration of nanoparticles increase viscosity of base oil which may result in high pressure drop in lubrication system. Increasing in viscosity will cause higher fuel consumption .It can also upset the balance of other additives such as detergent, anti-wear in the base oil itself as they are competing surfaces sites as mentioned by Thawhid et al. [15]. However, this project uses Al_20_3 with average molecular size of 70 nm with concentration of 0.5 vol % where it is proven by Abdollah et al. to be the optimal concentration for both Al_2O_3 and hBN nanoparticles when dispersed in SAE 15W-40 engine oil [16] .

2.4 Al_2O_3 and hBN tribological performance

Al_20_3 particle are hexagonally close-packed crystal materials resulting in excellent hardness, wear and heat resistances. This would make Al_2O_3 a good anti-wear properties. When Al_20_3 nanoparticle are dispersed in an engine oil, they can easily move into the worn area under the compressive stress of lubricating oil since their size are small and can squeeze through sliding contact and do not perturb the hydrodynamic regime. These anti-wear and friction reduction produce a lower friction coefficient and ultimately decrease the wear scar diameter [17], [18] . Furthermore, as there are friction between two contacting steel surface, there will be increase in flash temperature. As this occurs, the nanoparticles are absorbed chemically on the steel surface friction through hydroxyl and tribo-sinters on wear surface. This will greatly reduce steel on steel contact and as a load bearing areas [19] . This makes Al_20_3 a suitable candidate for improving EP properties for the reason being said. However, Al_20_3 nanoparticles tend to coagulate and agglomerate making it an undesirable traits. The agglomeration of Al_20_3 nanoparticles can be seen in the Figure 2.3 below.

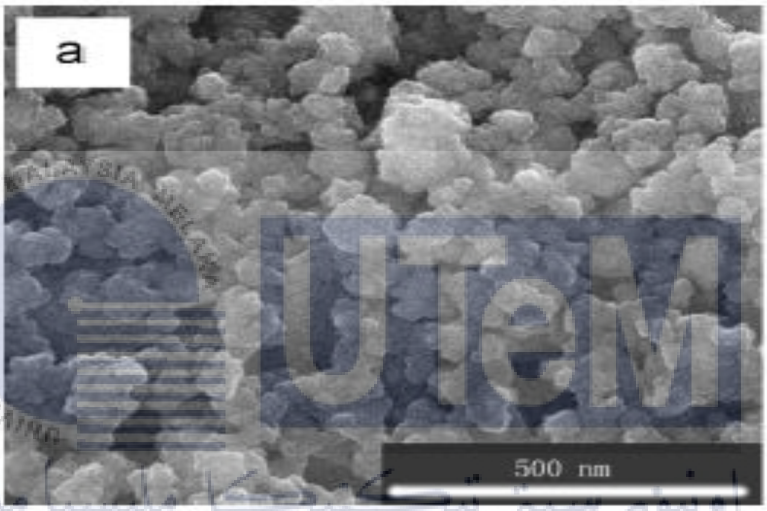

Figure 2.3 : The morphology of Al_20_3 which exhibits agglomeration

[Source : Luo et al. [18]]

Hexagonal Boron Nitride (hBN) in the other hand is also known as "white graphite" which have high thermal conductivity, stability and low surface energy which promises excellent behaviour such as good anti-wear and anti-friction. The hBN lamellar structure consists of a covalent bond where it can be found among the molecules. However, the bonding between the layers is held by weak Van Der Waals forces which are similar to graphite. The weak forces enable them to shears easily to lower the CoF of two contacting surfaces [20] . As reported by Abdollah et al. mineral engine oil when impregnated with 70nm of hBN nanoparticle with 0.5 vol % in concentration shows a significant results. Their research found that the hBN able to decelerate the seizure point on the contact surfaces, resulting higher EP and load carrying capability. The wear mode observed from the run are adhesive wear when lubricated with mineral oil compared to nano-oil where it reduces the severity of the adhesive wear [21]. hBN have lower rate of agglomeration making them much more desirable trait to have as an additive for engine oil as found by Abdollah et al. in their research on hBN effects on engine oil properties and as can be seen in Figure 2.4 below [22].

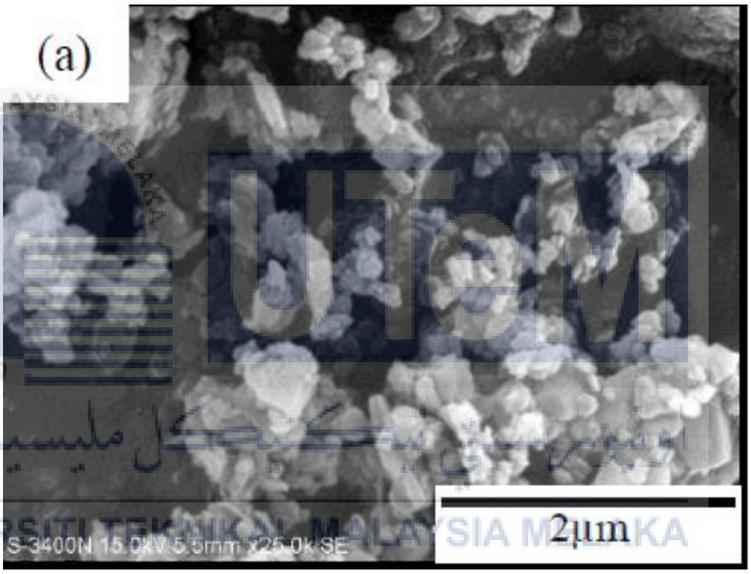

Figure 2.4: Morphology of hBN nanoparticles which exhibits uniform dispersion

[Source : Abdollah et al. [22]]

2.5 ASTM D 2596 and D2783

This standard is solely uses to determine the load carrying capability of lubricating fluids by two parameters which is load-wear index (Mean-hertz load) and weld point. The test is performed with one steel ball under a rotating load and three steel ball in stationary in form of a cradle. The steel ball are made from carbon-chromium steel with a diameter of 12.7mm and a HRC hardness ranging from 64-66 .The lower three balls are immersed with lubricant about 7-10 ml in volume which in this case SAE 15W-40 optimized with 0.5 vol % of Al_2O_3 and the base oil itself. The test parameter used in this run are rotating speed of 1770±60 rpm and 27± 8 °C in temperature and 10 seconds in duration. The tolerance value of the rpm and temperature are quite high because it can be used to differentiate lubricant having low, medium, and high level of EP properties. Preparation are needed where the balls need to be cleansed which utilized acetone as the solvent. The test are carried out with increment in loads after each run until welding occurs with load ranging from 196N to 1569N. One of the ball from each run should be measure to the nearest 0.01m scar diameter by placing them on a holder under the microscope. ASTM D 2783 also rules out that WSD exceeding 4mm are regarded as the weld point of the lubricant. The results obtain should follow the following graph plot which is the schematic Plot of WSD against the applied load as can be seen in Figure 2.5. The mean Hertz line is obtained through Eq. (2.1) and the load wear index can be calculate using Eq. (2.3)

$$Dh = 8.73\ x\ 10^{-2}(P)^{1/3} \tag{2.1}$$

where *Dh* is the Hertz diameter of the contact area, and *P* is the static applied load

$$\text{Corrected load, kgf} = LDh/X \tag{2.2}$$

where L is the applied load multiplied by lever arm ratio, Dh is the Hertz scar diameter ,mm and X is the average scar diameter

$$\text{Load –wear index, kgf} = A/10 \quad (2.3)$$

where A is the sum of the corrected load determined for the ten applied loads immediately before weld point

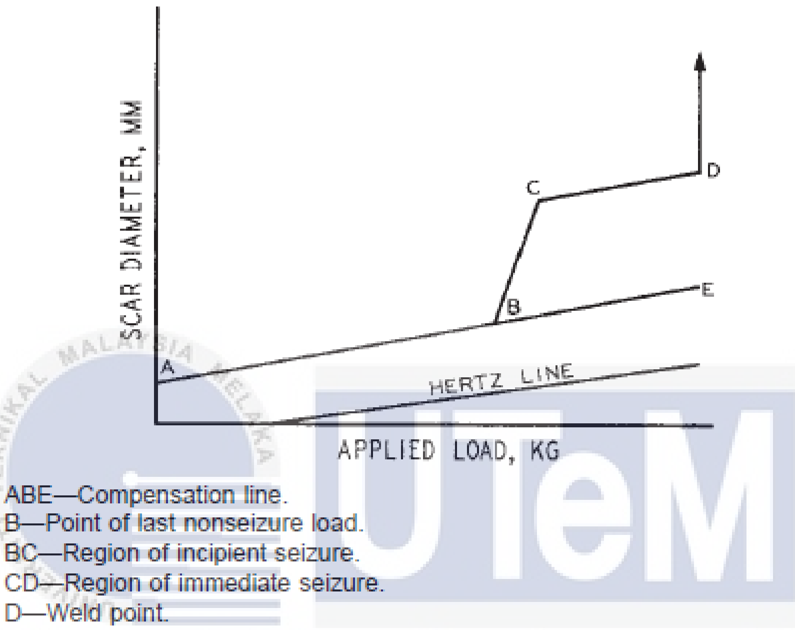

Figure 2.5: The schematic plot of WSD against load

[Source : ASTM International [23]]

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter discuss the methodology used to obtain the required data which is COF, WSD and Weld Load in order to study Al_20_3 tribological performance under extreme pressure. Characterisation from the tribometer test are done using Scanning Electron Microscopy (SEM) and Electron Dispersive X-Ray (EDX) Spectroscopy to observe the treated surfaces to be compared and differentiated. This study kicked off by following ASTM D 2596 and D 2783 standard test method for measurement of EP properties of the nanolubricants. This is crucial so that the experiment is carried out the correct way and the data obtain are legit. The overall flow of the experiment can be seen as in the Figure 3.1.

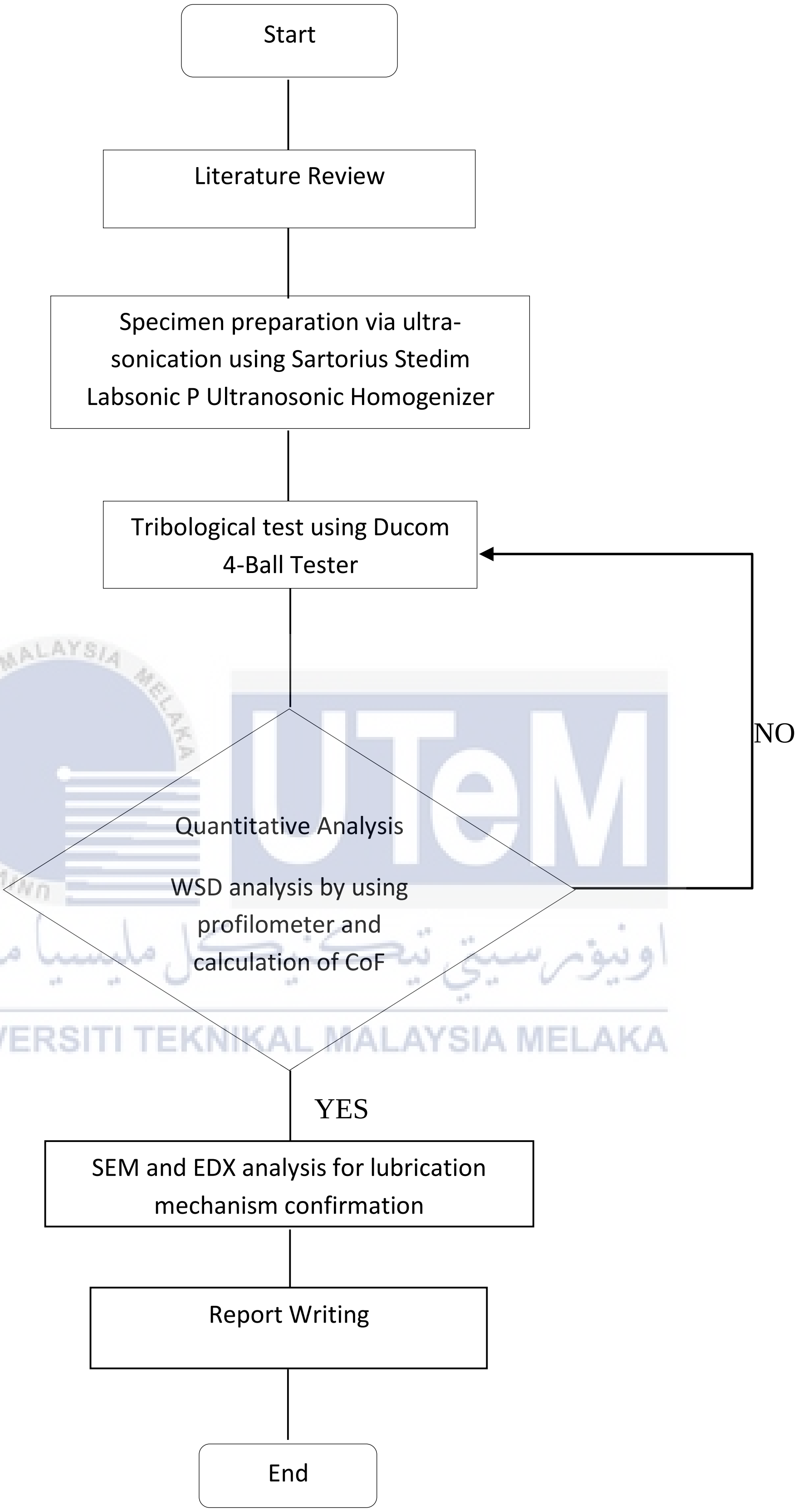

Figure 3.1 : Flow Chart of the project

3.2 General Experimental Setup

Figure 3.2 is illustrating the general setup for the characterization of lubricants which in our particular is EP properties. A dead weight is attached to the arm lever of the 4-Ball tester to apply load on the three bottom ball. The top ball are attached to the top collet and the three bottom ball are clamped together inside the ball pot. The lubricant are then poured inside the ball pot which simulates boundary lubrication regime. All the parameter discussed earlier in this report are keyed in via the Electronic Controller to meet the ASTM standards. Three parameter which is time, load and frictional torque are recorded by Winducom software which can be seen in Figure 3.3. A sensor is connected to the base of the ball pot which measures the frictional torque which enable us to calculate CoF.

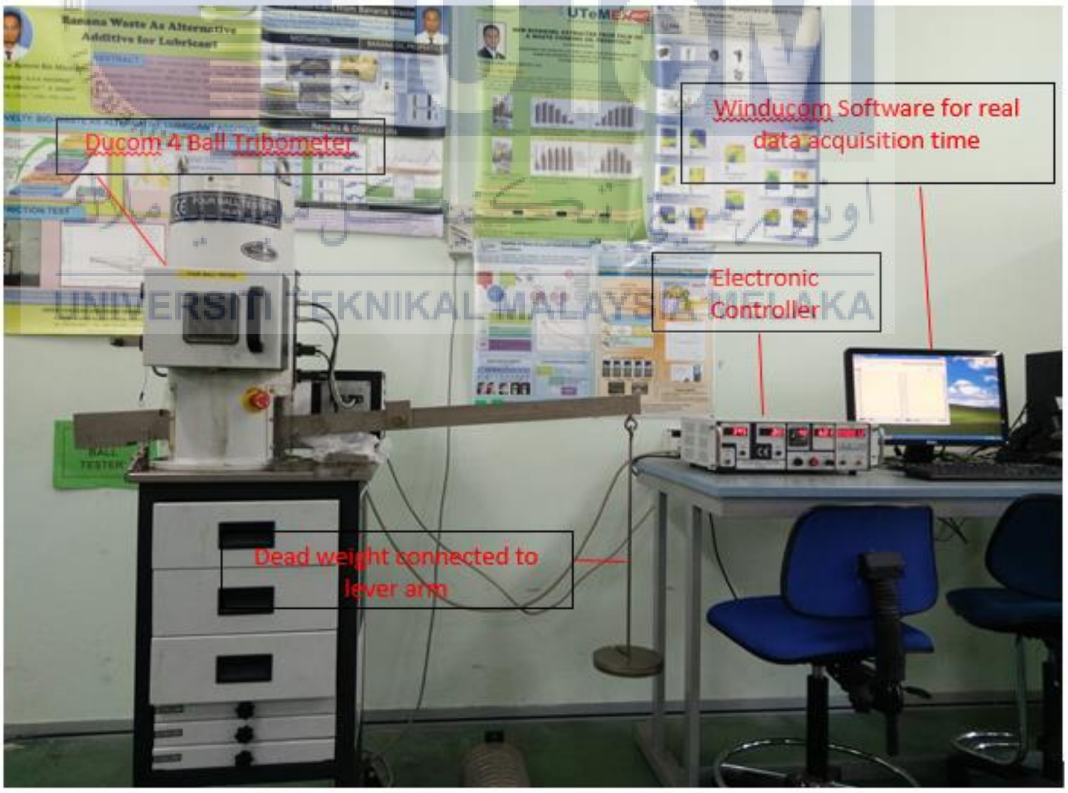

Figure 3.2 : Tribological experiment set up

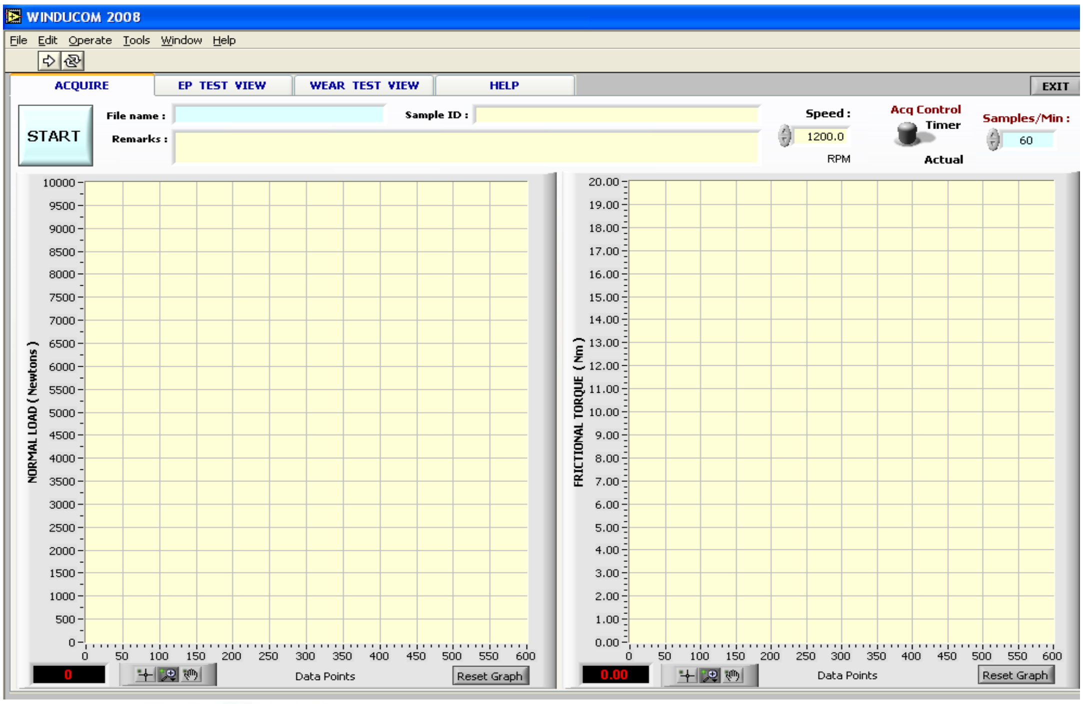

Figure 3.3 : Preview of the real time data acquisition software interface

3.3 Ultrasonic Homogenizer (Sample Preparation)

The main purpose of the ultrasonic homogenizer is to disperse the powder evenly in the base oil to prevent agglomeration of nanoparticles. This were done by using pulsating frequencies. The weighted powder are added into a glass bottle which contains 150 ml of base oil which are ready to undergo ultra-sonication. This process are done at 50% in amplitude with 0.5 active time interval for 80 minutes via Sartorious Stedim Labsonic Ultrasonic Homogenizer. The sonication duration had to be extended for 80 minutes as the powder does not dissolve easily which requires more stirring effort and time. The properties of the nanoparticles are also included in Table 1.

Figure 3.4 : The setting used for the sonication is 0.5 in cycle and 50 Hz in amplitude

Figure 3.5 : Sonication taking place

Table 3.1 : Properties of α-Al_2O_3 nano-powder

[Source : Auerkeri et al. [24]]

Properties	α-Al_2O_3 nano-powder
Appearance	White Powder
Chemical Formula	Al_2O_3
Purity	99.99%
Particle size	<100nm
Melting point	2045 ºC
Boiling point	2980 ºC
Density	3.97 g/cm^3

The sample are prepared by determining the volume of Al_2O_3 powder required to prepare a concentration of 0.5 vol % .

$$\left(\frac{v}{v}\right)\% = \frac{vome\ of solute\ (ml)}{volume\ of\ solution\ (ml)} x100$$

$$Density = \frac{mass(g)}{volume(ml)}$$

$$Volume\ = \frac{mass}{3.95}$$

$$0.5\% = \frac{mass(g)/3.95}{200\ ml} x100$$

$$mass\ (g) = 2.96\ g$$

$$mass\ of\ solute\ required\ to\ prepare\ 0.5\ volume\ \%\ is\ 2.96g$$

The procedure for sample preparation is as below:

1. The very first concern when handling this device is safety and electrical precautions. Handling this equipment requires proper PPE such as latex glove, eye and ear protection as there are risk of spillage and the ultra-sonic wave produced are high pitched.
2. The probe needs to be the chosen according to the total volume of the specimen. In this case, the specimen volume is 150ml and the diameter of the probe used is 7mm with approximately 80mm in length.
3. The probe are threaded into the shaft and tighten using 14mm and 19mm wrench to avoid the probe from unthreading itself.
4. The glass bottle are submerged into a beaker filled with water to avoid the glass bottle from overheating during sonication.
5. Place the beaker on the base and rise the base until the tip of the probe are half-submerged into the specimen.
6. Ensure the tip of the probe are at the centre of the glass bottle so that the seizure location are correct and the sonication are done successfully.
7. Tighten the knob of the base to ensure that glass bottle are held in place
8. Close the door of the cabinet avoid exposure of aerosols and turn on the switch. The sonication are done for 80 minutes
9. Once finished, the probes need to be rinsed using acetone and cleansed using tissue paper

3.4 Ultrasonic bath

Before the SKF carbon-chromium steel ball can be use for the 4 ball tester, they are required to undergo ultrasonic bath. This is to remove the contaminant and grease which SKF used to prevent rust and it is done using GT Sonic Ultrasonic Bath. The cleaning solution used is acetone and the detailed breakdown of the procedure is as below

1. 4 of 12.7mm carbon-chromium steel ball is placed inside a beaker by using a spatula or tweezers
2. Before handling acetone, PPE such as gloves and eye protection are needed to be worn all time
3. Acetone are poured in the beaker until they are fully submerged
4. Place the beaker inside the ultrasonic bath and the lid are placed on top of the bath
5. The bathing time of the ball are set for 10 minutes.
6. Once the bathing are done, the ball are cleansed thoroughly with tissue paper before being used for the tribometer test.

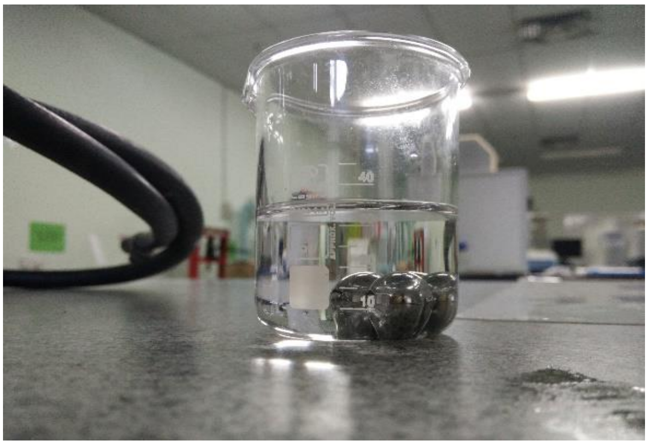

Figure 3.6 : SKF Steel ball immersed in acetone

Figure 3.7 : Steel ball being clean with the ultrasonic bath machine

3.5 Four Ball Tester (Tribological Test)

The interest of this study is the tribological performance of Al_2O_3 under EP in order to determine the load carrying capabilities under high load applications. The 4-Ball Tester operates in rolling motion where a top ball rotates on three bottom ball which lubricated in boundary lubrication regime. The load carrying capabilities are measured in three steps which is

(i) The last non-seizure load which is the highest applied load where lubricant still protecting the 4 ball from direct contact.

(ii) Seizure region where the lubricant no longer protecting the balls and direct contact of the ball occurs.

(iii) Welding load is the catastrophic load where the balls suffers heavy damage. The load from these 3 parameters can be used to calculate load wear index (LWI) which will provide numerical value to further analyse Al_2O_3 performance and the safe load it can takes.

The test parameter of this experiment and the mechanical properties of the steel ball are tabulated in Table 2 and Table 3 respectively. The working principle of the tribometer can be seen as in Figure 3.9 and it is already discussed in previous chapter.

Table 3.2 : ASTM Standards for Extreme Pressure test

Standard	Load (N)	Duration (sec)	Temperature °C	Speed (rpm)
ASTM D 2596 and D 2783	196-1569	10 sec AT EACH LOAD	27 ± 8	1770 ±60

Table 3.3 : The mechanical properties of the carbon chromium steel ball manufactured by SKF

[Source : SKF USA Inc [25]]

Properties		Carbon-chromium steel ball
Hardness,	HRC	61
Density	g/cm^3	7.79
Surface Roughness	µm	0.022
Diameter	mm	12.7

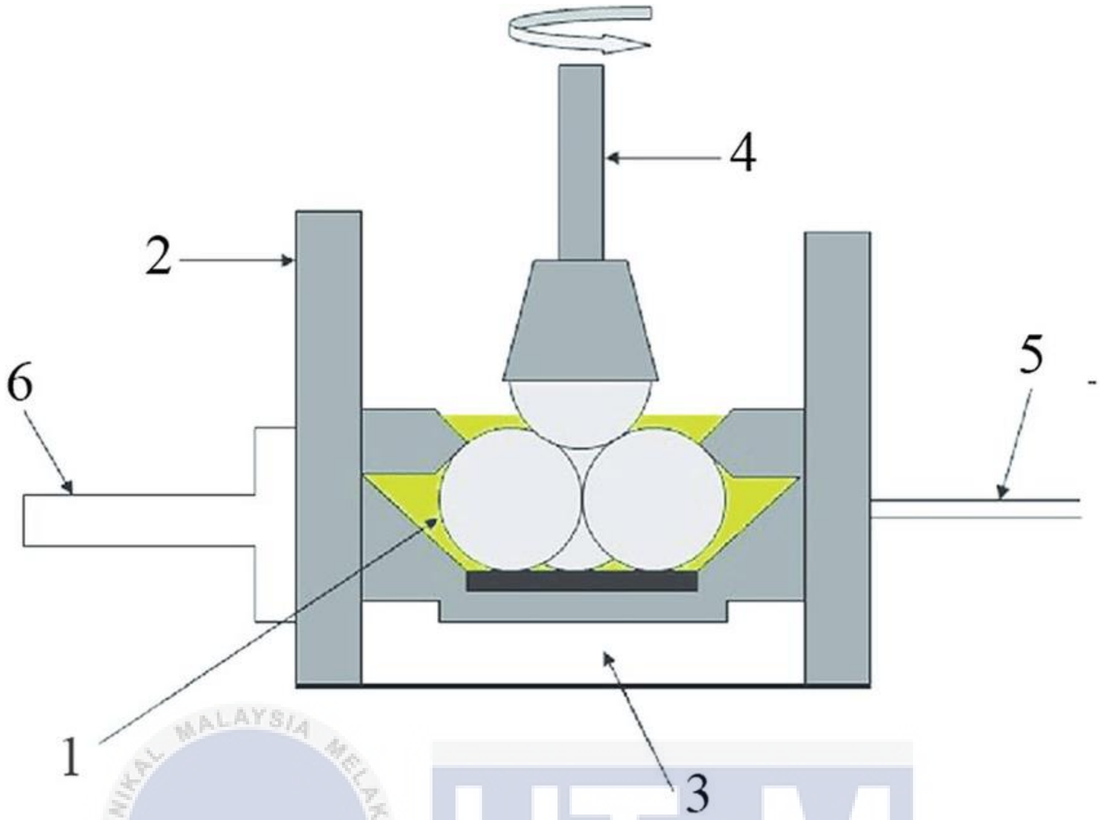

Figure 3.9: The schematic diagram of the 4-Ball tester

[Source : Leang et al. [26]]

The step by step for the test are as follow:

1. The ball pot are cleansed with acetone and wiped with tissue paper to remove contaminant,debris and oil from previous run
2. The ball locking ring are inserted into the ball pot and the three bottom ball placed inside it
3. The locking nut are threaded in and tightened using torque wrench with torque rating of 40-50 N.m
4. The lubricant are poured in at about where the bottom ball are almost submerged to achieve the boundary lubrication condition.

5. The ball are inserted into the collet and secured by pressing the ball onto the collet master. This is crucial to avoid the ball from vibrating and spinning uncontrollably during the run
6. The collet are inserted into the spindle and are align by slotting in the collet into the slot.
7. The ball pot are transferred into the equipment and placed on the base. The ball pot needs to be align perfectly with the best to ensure stability and safety during extreme pressure environment.
8. The sensor are connected to the ball pot and the weight hanger are attached at the weight arm.Align the pot with the frictional torque sensor
9. The dead weight are added to the weight hanger and the balancer are used to balance out the weight.
10. The test parameter are keyed in into the electronic controller which in 10s in duration, 27±8 °C in temperature and 1770±60 rpm in speed.
11. The software are runned continuously and sequentially with the actual test to collect real time data
12. Once the run finished,the arm lever are lifted and stacked against the stopper and the dead weight are removed.
13. The sensor connected to the ball pot are removed and the locking nut are removed using the torque wrench. The ball are removed and immersed in acetone to undergo bathing process as dictated in Methodolgy 3.4
14. Remove the top ball from the collect by pushing the pin of the collect master against the ball.
15. Clean all equipment with acetone and repeat steps 1-14 for the next run

3.6 Quantitative analysis

After all the tribological test and the WSD measurement are carried out, the results obtained needs to be analyzed thoroughly and systematically. There are few equation that needs to be taken account in in order to analyze the EP properties of the base oil and Al_2O_3 and hBN. All the calculation shall be done using the formula stated in Chapter 2, Section 2.5 which composed of hertz line diameter, corrected load and load wear index.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Al_2O_3 nano-particles extreme pressure properties

One of the two objectives of this study is to study the extreme pressure properties of Al_2O_3 nanoparticles. It is best demonstrated in form of graphical analysis for easier understanding and clearer information

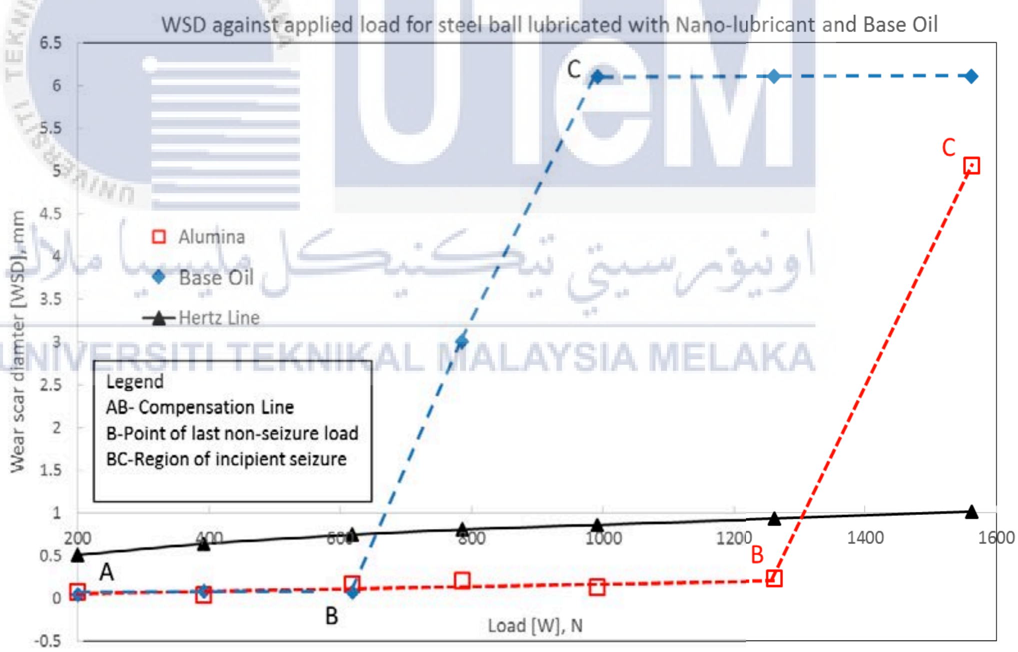

Figure 4.1 : WSD against applied load

For the base oil, as the load increases from 200N indicated as point A to point B with load of 618N, the increment of WSD has small variation and almost similar to increment of WSD of the nano-oil. However, as the load increases from 618N, the WSD for the base oil increases roughly from 0.075mm to 3.00mm. From the analysis that has been done, the point of the last non-seizure load indicated as “B” of the nano-oil is at 1236N with WSD approximately 0.228mm compared to the base oil which is at 618N with WSD of 0.075mm. This indicates that the nano-oil containing Al elements has improved the load carrying capability of the base oil. As the load increased from the point of the last non-seizure load, the WSD of the base oil increase rapidly and enters region of incipient seizure. This is an indication that the base oil is no longer lubricating the steel ball and the steel ball is now experiencing direct metal to metal contact which occurs from 981N to 1569N. This explains why the WSD of the base oil increased rapidly and large in diameter. Severe adhesive wear were also spotted causing seizure of the steel ball. ASTM D 2783 standards outline that if the WSD and load wear index that exceed 4mm is the weld point. For the nano-oil, even after the point of the last non-seizure load, the WSD remains minimal until it reaches 1236N. After it went beyond 1236N, the WSD of the steel ball lubricated with nano-oil increase rapidly and enters the region of incipient seizure up to 1569N in load. The nano-oil able to reduce the severity of the adhesive wear significantly compared to the base oil and increases the weld point form 981N to 1596N.

The improved load carrying capability are further analysed using JEOL JSM-6010 PLUS Scanning Electron Microscope. From the analysis, Al signal is spotted on the clean surface although the signal is somewhat weaker compared to the other element where the same exact result were also obtained by Luo et.al [18]. This suggest that Al element indeed deposited onto the worn surface. However, the deposited Al solid might be too small to be count making their weight percentage unattainable. The SEM applied voltage are also varied are few time to penetrate the sample surface without damaging it to count the Al element weight percentage which also failed. Therefore, mapping process were done to validate the presence of Al element that contribute to the improvement of the base oil properties. The analysed worn surface are as in Figure 4.2 and the spectrum result are as in Figure 4.3. The mapping process then managed to be performed and the presence of Al element are spotted on the worn area as can be seen in Figure 4.4(a) and Figure 4.4(b).

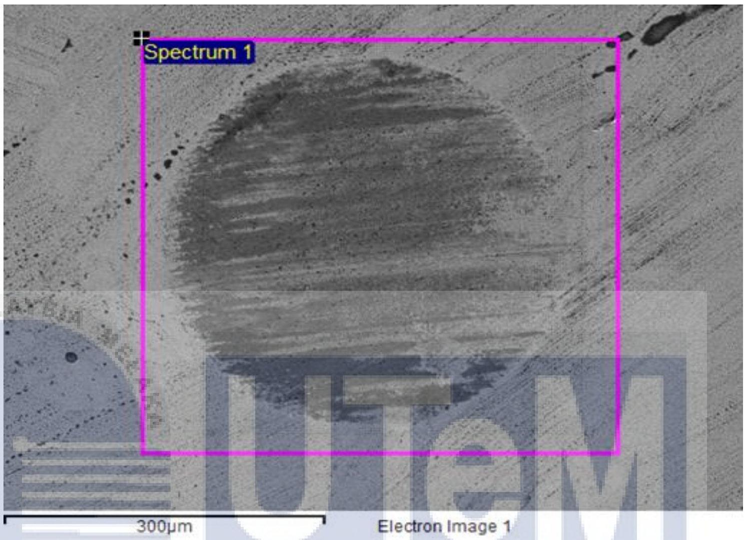

Figure 4.2 : SEM images of the worn surface lubricated with Nano-oil

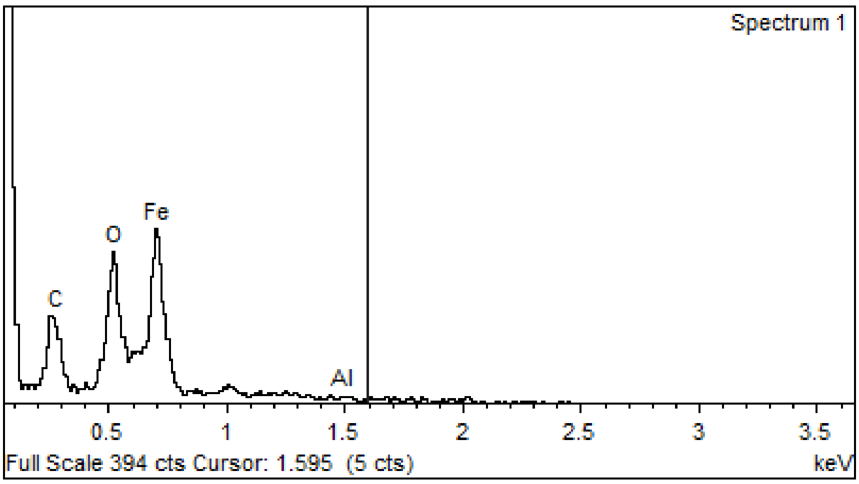

Figure 4.3 : EDX spectrum of the worn surface

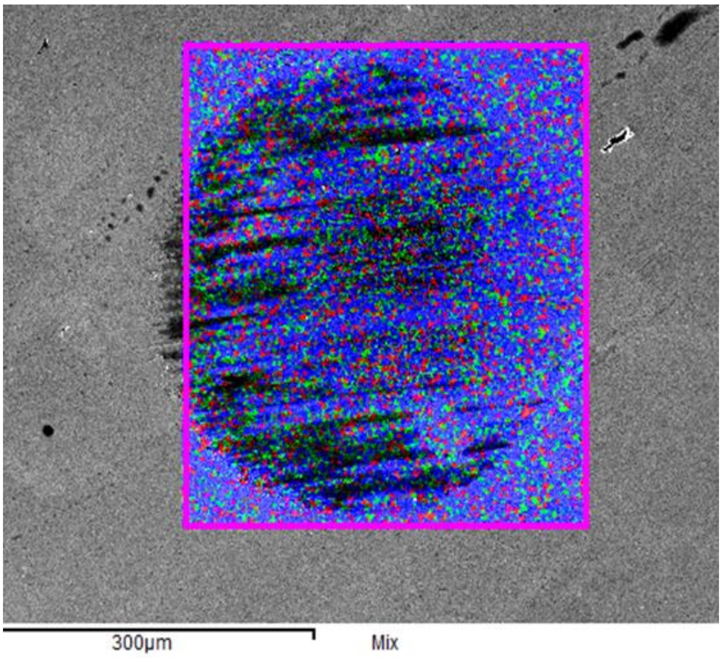

Figure 4.4 (a) : Overall map of the worn surface

Figure 4.4 (b) : Mapping for Al element on the worn surface

The lubricating mechanism that takes place in improving base oil properties is the most important thing and it varies with the base oil used and the nanoparticles itself. There are 4 possible lubricating mechanism involved in the improvement of the load carrying capability and non-seizure load. The first one is protective film or also known as tribofilm formation. Al_2O_3 is an oxide particle that form protective layer which arises from tribo-sinter reaction where some of the oxide particles which are in contact with rubbing surfaces in which our cases are the steel ball, are compacted and sintered forming tribofilms as reported by Luo et al. [18]. These tribofilms contains Al complex that helps to reduce severe wear which in turn improving the load carrying capability of the nanolubricant. The protective layer effect are further analysed by mapping process on the worn surface. This is in agreement of other researcher such as Verma et al. [27] where nano MoS_2-based lubricant forms a durable thin tribofilm containing Mo, S and P elements effectively reduce severe wear, friction, and seizure. Hernȧndez et al. [19] discovered NiCrBSi lubricated with CuO dispersed in polyalphaolefine (PAO6) able to show reduction in friction and the nanoparticle show significant tribosinterizations. He discovered that increased in temperature resultant from friction makes the nanoparticles chemically absorbed on the worn surface through hydroxyl and tribo-sinter reaction which bears the load to reduce metal contact which are then confirm through Energy Dispersive X-ray Sprectroscopy (EDS) and X-Ray Photoelectron Spectroscopy (XPS). However, the tribofilm formation in this project are unable to be confirmed as the surface characterization analysis are not acquired since they are not available

The second lubricating mechanism that involved are mending effect. Mending effect can be termed as deposition of solid nanoparticle onto the grooves or on the worn surface which reduce surface roughness. As the Al element deposited onto the metal surface, it forms a new protection surface that inhibits metal contact. This give the metal surface better wear protection and hence, higher load carrying capabilities and lower CoF. Since Al_2O_3 made up of hexagonal crystalline structure, it gives them an excellent anti-wear properties thanks to their hardness. With their excellent hardness, the said protection surface it form would allow them to be more durable which is highly desirable especially in the critical area of an engine such as piston ring liner and journal bearing of the crankshaft. This is in agreement with Wu et al.[28] as he discovered that TiO_2 nanoparticles used in water based lubricant able to fill in the grooves, cracks and gap of the work piece that effectively reduce surface roughness and thus lower CoF. Giu et al. [28] which uses 500SN base oil with 0.1 wt% Cu nanoparticles exhibits stellar mending effect where they employed a pin on disk experiment. From his finding, the mending effect contributes to flat, smoother and shallower groove on the pin compared to the pin lubricated with the base oil itself. The mending effect of Al are also investigated using SEM analysis which are presented in Figure 4.2 and Figure 4.3 respectively.

The third lubricating mechanism involved are ball bearing effect. Al_2O_3 are spherical in shape enabling them to play the ball bearing effects which effectively change sliding friction into rolling friction. Rolling friction effectively reduce the CoF of the contact surface which makes all rotation takes place smoothly contributing to less and smoother wear. Al_2O_3 nanoparticles are also small enough at 70 nm which enable them to align themselves at extreme pressure area which then separate two metal surface. Figure 4.5 below shows the differences in CoF, μ of the steel ball lubricated with nano-oil and base oil at the same load of 391N and 981N. It can be observed that the CoF of the steel ball lubricated with nano-oil is significantly lower compared to base oil at same load. The one lubricated with nano-oil also shows a smoother trend where they able to maintain a stable friction coefficient over the period of the test. This contributes in excellent extreme pressure properties and load carrying capability. Figure 4.6 shows the average reduction of CoF between Al_2O_3 enriched nano-oil against base oil where it shows the significance of the spherical nanoparticles by lowering it up to 48.95%.

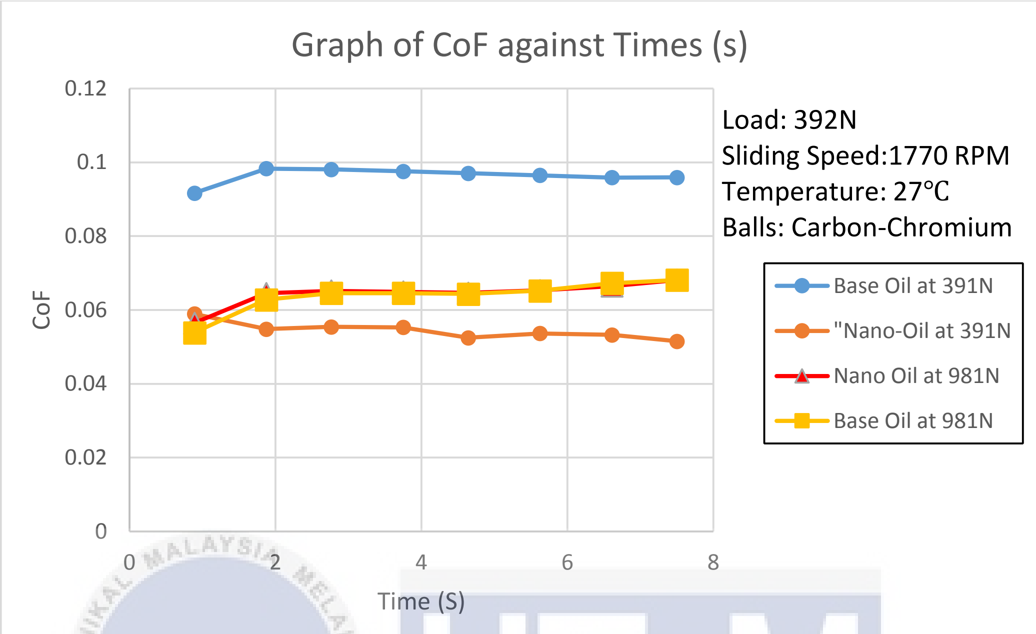

Figure 4.5 : Graph of CoF against Time(s) for steel ball lubricated with Al_2O_3 nano-oil and base oil.

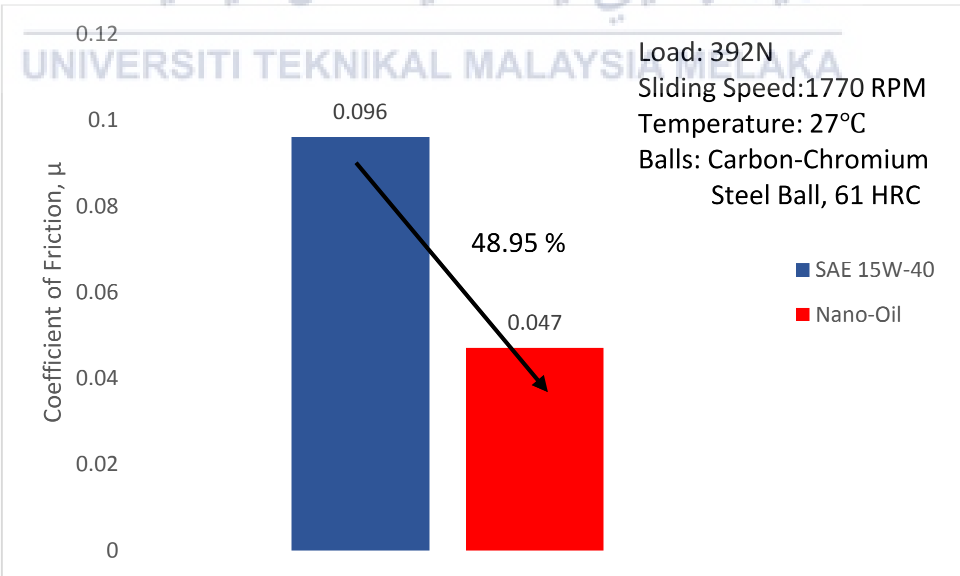

Figure 4.6 : Coefficient of friction reduction, μ between Al_2O_3 enriched nano-oil against base oil

The fourth lubricating mechanism that might involve are polishing effects. Polishing effect are also known as smoothing effect where these nanoparticles fill up the gaps of rough surface. As the surface roughness is reduced, it results in increased tribological performance in terms of CoF as the surface are now much more smoother and lower as mentioned by Kheireddin et al. [1]. However, this effect in still uncertain as it requires surface topography analysis tools such as Atomic Force Microscopy (AFM)

4.2 Identifying the best nano-particles in diesel engine oil.

In this project, two nanoparticle are used which is Al_2O_3 and hBN with similar size dispersed in diesel engine oil. Therefore, we need to identify the best nanoparticles to be used in regards to improve the EP properties of the base oil.

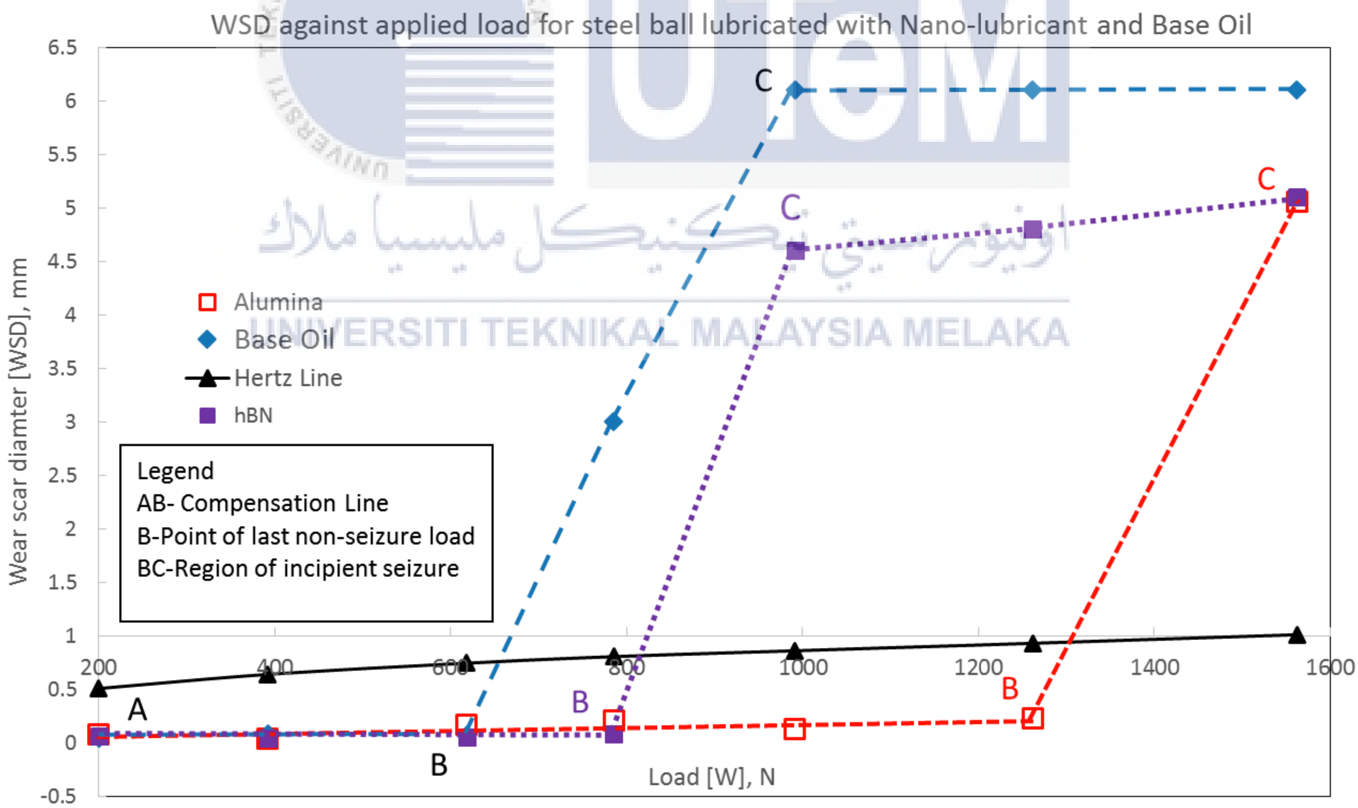

Figure 4.7 : WSD against applied load for steel ball lubricated with Nano-Oil and Base Oil

Both Al_2O_3 and hBN nanoparticle plays a major role in improving the extreme pressure properties of the base oil. One of the most remarkable improvement is in the last non-seizure load, where both of the nanoparticles increase it from 618N to 786N for hBN and 1236N for Al_2O_3 with WSD of 0.08mm and 0.228mm respectively. Both nanoparticles also decelerate the incipient seizure load by lowering them at a significant rate. The base oil already experience immediate seizure and weld point at 3.00mm with load from 981N to 1569N whereas both base oil added with nanoparticle still able to maintain protection and hence increasing the extreme pressure of the base oil. The weld point of the hBN nano-oil are already started at 981N and it increases from there all the way to 1596N with WSD of 5.1mm while for Al_2O_3 nano-oil, the weld point are at 1596N with WSD of 5.058mm This is because, both nanoparticle introduce a new lubricating mechanism enhancing the said aspects as can be seen in Figure 4.7 above. Al_2O_3 and hBN displays the same lubricating mechanism but there is a slight difference on how they operate since they have different properties.

First and foremost are mending effect where the solid nanoparticle are deposited onto the grooves or on the worn surface which reduce surface roughness as in Figure 4.8 below. The ball surface are irregular which can only be seen at microscopic level where theoretically will have high CoF and causes greater wear. As these nanoparticles enters the contact area under extreme pressure and bears the load, they are crushed and fills up the irregular surface of the asperities. Therefore, they self-mend onto the surface and effectively increase the surface roughness. This in turn would greatly reduce the WSD and friction coefficient for the given applied load resulting in shallower plow furrow. Since hBN are generally soft, they have the characteristic of shock-absorbing which can easily "sacrifice" itself to minimize direct contacts of metal. Even though soft nanoparticles are more preferable since they are better at absorbing shock, there is a trade-off where they unable to sustain higher extreme pressure as Al_2O_3. For Al_2O_3 nanoparticles, they have excellent hardness and wear properties which explains why Al_2O_3 nanoparticles significantly improves the extreme pressure of the base oil due to their hexagonal crystalline structure. Because of their excellent hardness and anti-wear properties, they can sustain higher extreme pressure and prevent direct contact of metal much better compared to hBN nanoparticles. This are in agreement with Avinash et al. [29] where medium sized Al_2O_3 ranging from 70nm-80nm provides the most valuable mending effect which suits this project finding.

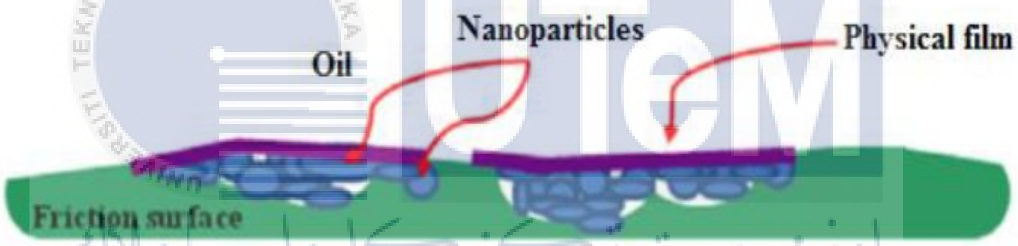

Figure 4.8 : Illustration of mending effect where nanoparticles fills up the irregular surface on worn surface

[Source : Kheireddin et al. [1]]

The second lubricating mechanism are ball bearing effects. Both nanoparticles are spherical in shape enabling them to play the ball bearing effects which effectively change sliding friction into rolling friction. Rolling friction effectively reduce the CoF of the contact surface which makes all rotation takes place smoothly contributing to less wear. Figure 4.9 can illustrate on how there spherical nanoparticle work where they mobilize at high speed and align themselves along the sliding direction of the worn surface. Both nanoparticles are at the optimum size of 60-70nm which can contribute noticeable rolling and are crushed between asperities to separate the asperities minimizing CoF [29]. Low friction means greater wear deceleration and higher load carrying capability. This is highly desirable since the base oil only contains oil film with no solid particles to enhance it lubricating mechanism. The rolling effect of Al_2O_3 have been discussed in Paragraph 4.1, Figure 4.5 and 4.6. However, a detailed and thorough investigation regarding to the ball bearing effect of hBN nanoparticles needs to be done so that it can be compared further with the ball bearing effect of Al_2O_3 nanoparticles.

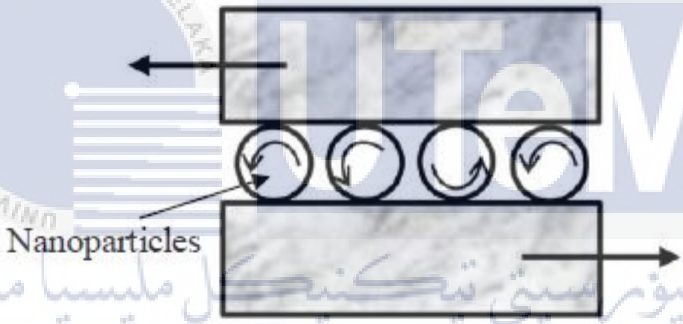

Figure 4.9 : Illustration of ball bearing mechanism

[Source : Abdollah et al. [16]]

The third lubricating mechanism that involved in improving the base oil extreme pressure properties which is protective film. hBN forms a Boron complex with low shear strength film. This means that this protective film can easily shear off to form sacrificial oil layer which help separate metal-to-metal contact as it is consumed to provide the protection. hBN nanoparticle dispersed in SAE 15W-40 diesel engine oil produced a tribofilm containing B-complex are able to increase the point of last non-seizure load of the base oil. This is in agreement with Ji et al [30] where he discovered that tri(hydroxymethyl)propane ester containing B element dispersed in rapeseed oil able to increase the point of last non-seizure load of the rapeseed oil at 981 N. On the other hand, Al_2O_3 is an oxide particle that form protective layer which arises from tribo-sinter reaction where some of the oxide particles that are in contact with the rubbing surfaces in which our cases are the steel ball, are compacted and sintered forming tribofilms. The tribofilms formed are impregnable as it arises from the reaction of base oil, nanoparticles and existing additive in the base oil itself. The said tribofilm produced able to resist oxidation and wear of the steel ball. helps to reduce severe wear which in turn improving the load carrying capability of the nano-oil. However, Al_2O_3 protective film effects need to be validate via SEM analysis as it is not acquired in this project. Figure 4.10 below demonstrates the formation of tribofilm where it plays two vital role as they inhibit crack propagation and surface protection as explained by Gulzar et al [31] .

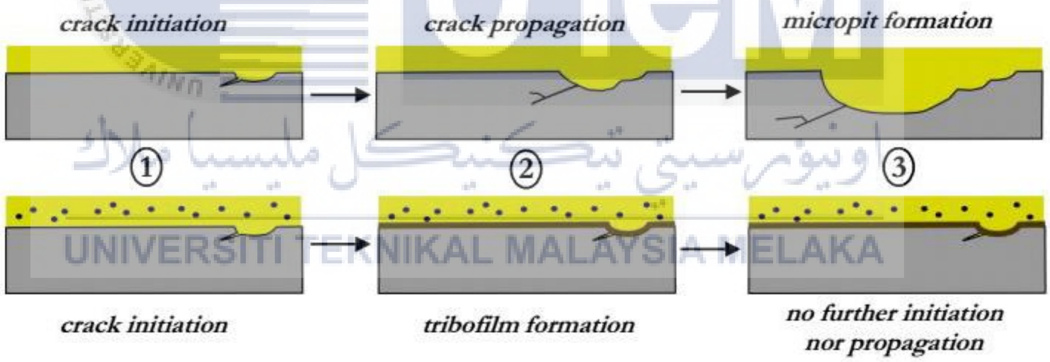

Figure 4.10 : Illustration of tribofilm formation

[Source : Rabaso et al. [32]]

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The research and development on reducing friction and wear has been the main focus in tribology field. The increase demand in higher extreme pressure lubrication has driven the passion to find the suitable nano-additive to enhance such criteria. With the dispersion of suitable nano-additive, enhancement of the key lubricating properties on boundary lubrication can lead to more enhancement on lubricating properties that improve the overall performance of an IC engine in terms of BSFC, extended load interval and longer engine lifespan. This is due to the reduction in CoF which leads to lesser wear and more anti-wear properties which is highly desirable in extreme condition of engine operations. However, even with extensive research in EP properties, there are still huge gap of knowledge and application of nanolubricant in the real world. This study was conducted to provide a form of sharing and addition of knowledge and information to the tribology field and the future researcher in particular. Therefore, this study comes up with the following conclusions

a) The load carrying capability of the base oil in boundary lubrication regime can be enhanced with the addition of Al_2O_3 nanoparticles at an optimum concentration. This is proven where the point of last non-seizure load for Al_2O_3 enhanced base oil as it is increased from 618 N to 1236 N. The addition of Al_2O_3 nanoparticle also reduce the severity of adhesive wear observed when the ball are lubricated with the base oil itself. This is due to the lubricating mechanism involved discussed before which is protective film, ball bearing effect and mending effect. Therefore, the first objective of this study has been achieved as we obtained the EP properties of Al_2O_3 enriched base oil.

b) The best nanoparticle to be used in diesel engine oil is Al_2O_3 nanoparticles. This is because, the load carrying capability are improved almost twice the base oil. The point of last non-seizure load for hBN nanoparticle are 786N, 1236N for Al_2O_3 nanoparticle and 618N for the base oil itself. Both of these nanoparticle are also able to reduce the severity of the adhesive wear obtained from the steel ball lubricated with the base oil which is very promising. With that being said, the second objective of this project has been achieved.

5.2 Recommendation

For future research, it is recommended that all the base oil that is being used to be the same brand and viscosity. Different oil brand will have different blend of additive and key lubricating properties which might play a noise factor contributing to varying data accuracy. Different oil brand also might have already have extreme pressure and anti-wear additive in which of further addition of nano-additive might not play their role to their potential. The nano-oil also should be stabilized by the usage of surfactant such as oleic acid to pro-long the time taken for sedimentation to develop and kept the nanoparticles remains dispersed in the engine oil. The extensive effect of nano-additive also should be investigated further by testing the lubricant into a real engine. However, the sedimentation problem needs to be addressed first before the real application takes place to prevent clogging of oil gallery of an engine. It would be highly desirable to apply all the knowledge obtain in tribology field for the benefits of consumer in having extended protection of the critical area of an engine as well as enjoy greater fuel efficiency. An experiment which employs Taguchi Method also should be considered so that the quality of the experiment increased significantly.

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APPENDICES

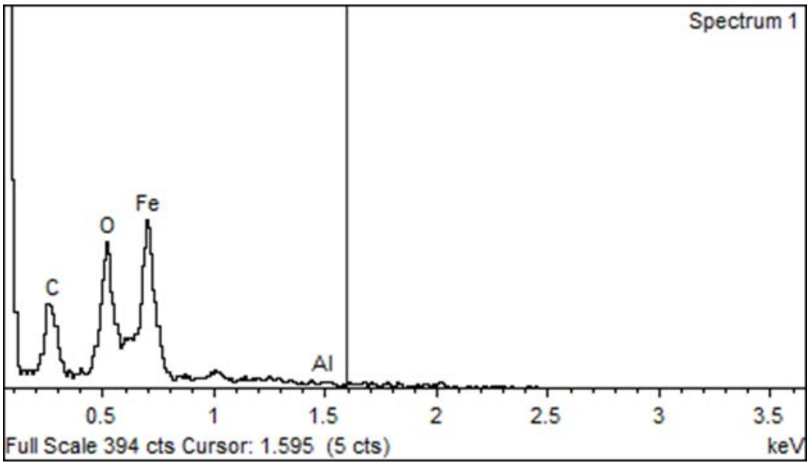
Appendix A1: Gant Chart for PSM 1

Activity	Semester 1 Session 2018/2019														
	Week														
PSM 1	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Project Planning															
Chapter 1: Introduction															
Chapter 2: Literature Review															
Chapter 3: Methodology															
Chapter 4:Prelimenary Results															
Progress Report Submission															
Sample Preparation 0.5 vol % of Al_2O_3															
Experiment Ducom 4-Ball Tester															
Analysis Wear Scar Diameter															
Report Submission															

Appendix A2: Gant Chart for PSM 2

Activity	Semester 2 Session 2018/2019																	
	Week																	
PSM 2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
SEM and EDX analysis																		
Literature Review																		
Chapter 4 : Results and Discussion																		
Chapter 5 : Conclusion and Recommendation																		
Progress Report Submission																		
Draft Final Report Submission																		
Final Report Correction																		
Hardbound Submission																		

Appendix B : SEM Results



Element	Weight%	Atomic%
C K	7.04	20.49
O K	13.65	29.83
Al K	0.00	0.00
Fe L	79.32	49.67
Totals	100.00	