ANALYSIS OF GREASE DEGRADATION UNDER ELASTOHYDRODYNAMIC LUBRICANT

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DECLARATION

This work has not previously been accepted in substance for any degree and is not being concurrently submitted in candidature for any degree.

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LIST OF ABBREVATIONS

ASTM	American Society for Testing and Material
AW	Anti Wear
COF	Coefficient of Friciton / Friction Of Coefficient
Cu	Copper
DLC	Diamond-like Carbon
DSC	Differential Scanning Calorimetry
EDX	Energy Dispersive X-Ray
EHL	Elastohydrodynamic Lubricant
EP	Extreme Pressure
FESEM	Field Emission Scanning Electron Microscope
FTIR	Fourier-Transform Infrared Spectroscopy
IFM	Infinite Focus Microscope
MoS2	Molybdenum Disulfide
PDSC	Pressure Differential Scanning Calorimeter
Ra	Surface Roughness
SEM	Scanning Electron Microscope
TEM	Transmission Electron Microscopy
TGA	Thermogravimetric Analyser
WSD	Wear Scar Diameter

ABSTRAK

Prestasi gris adalah penting kerana ia boleh mempengaruhi geseran dan haus komponen yang dilincirkan. Tujuan kajian ini adalah untuk menyiasat prestasi dan degradasi gris dalam aplikasi sesentuh bergolek. Dalam kajian ini, empat jenis gris telah dikaji iaitu minyak sawit dengan gris nanozarah Cu, minyak sawit dengan zarah mikro MoS_2, gris mineral SKF LGGB 2/0.4 dan gris makanan BIOGREASE Gred Marlin-9. Piawaian ASTM D2266 digunakan untuk mengkaji ciri pencegahan haus gris pelincir. Prestasi gris pada suhu tinggi dan beban dengan nilai 100 °C dan 100kg juga dinilai. Mesin Four Ball Tester DUCOM, mesin Hitachi TM1000-Tabletop Microscope, mesin Hitachi S-3400N SEM, mesin Alicona IFM-Optical 3D Surface Morphology dan Thermogravimetric Analyzer Pyris 1 (Perkin Elmer) telah digunakan dalam kajian ini untuk menganalisis prestasi gris dan degradasi. Penemuan menunjukkan bahawa prestasi gris minyak sawit dengan bahan tambahan adalah setanding dengan gris mineral komersial. Pada ujian suhu dan beban yang tinggi, minyak sawit dengan bahan tambahan Cu dan MoS_2 menunjukkan nilai COF yang rendah dan stabil, dan ini menunjukkan bahawa tiada degradasi berlaku pada gris apabila beroperasi pada suhu dan beban yang tinggi. Gris ini juga menunjukkan prestasi terbaik pada aplikasi beban tinggi dengan menunjukkan hasil kekasaran permukaan dan profil kedalaman yang rendah. LGGB mempunyai prestasi tribologi terbaik dalam semua aspek dan ini tidak dapat dinafikan kerana ia adalah gris komersial yang telah digunakan di seluruh dunia. Gris Gred Makanan mempunyai prestasi yang paling teruk kerana gris jenis ini digunakan secara meluas dalam operasi pengendalian makanan. Untuk mengelakkan bahaya kepada manusia, ramuan formulasi mestilah selamat kepada manusia tetapi pada masa yang sama boleh melindungi mesin.

ABSTRACT

The performance of grease is important as it may influence the friction and wear of the lubricated components. The purpose of this study is to investigate the performance and degradation of the grease in rolling contact applications. In this study, four types of grease has been studied, there are palm oil with Cu nanoparticle grease, palm oil with MoS_2 micro particle, SKF LGGB 2/0.4 mineral Grease and BIOGREASE Food Grade Grease Marlin-9. ASTM D2266 Standard were used to study the wear preventive characteristic of the lubricating grease. The grease performance at high temperature and load with the value of 100℃ and 100kg are also evaluated. DUCOM Four-Ball Tester Machine, Hitachi TM1000-Tabletop Microscope machine, Hitachi S-3400N SEM, Alicona IFM-Optical 3D surface metrology and Thermogravimetric Analyzer Pyris 1 (Perkin Elmer) were used in this study to analyse the grease performance and degradation. The finiding shown that the performance of the palm oil grease with additive is comparable to the commercial mineral grease. At high temperature and load test, the palm oil with Cu and MoS_2 additive show low and stable COF values, and this indicates that no degradation occurs to the grease when operating at high temperature and load. These grease also show the best performance at high load application with a small roughness and wear depth profile. LGGB has the best tribology perfomance in all aspect and this is undeniably because it a commercial grease that has been used all over the world. Food Grade has the worst performance because this type of grease is widely used in the food line operation. To prevent harm to human, the formulation ingredients must be safe to human but at the same time can protect the machine.

CHAPTER 1

INTRODUCTION

1.1 Introduction

According to the American Society for Testing and Material (ASTM), lubricating grease is "a solid to a semi-fluid product of dispersion of a thickening agent in liquid lubricant." Few individuals are aware of the distinctions between oil and grease, despite the fact that most people are familiar with the terms. Some circumstances call for the use of oil, while others call for the use of grease. Because oil has the ability to transmit heat from one body of oil to another, which is then circulated through a heat exchanger, it is employed in any application that generates a lot of heat.

In contrast, grease is used to lubricate machinery that is only oiled occasionally since lubricating oil would not adhere to the machine's surfaces. In order to keep out water and incompressible materials, it also acts as a sealer. In addition, grease can be utilized on moving machines that are operated in closed environments for extended periods of time and under challenging or a typical working conditions. Grease is a semisolid lubricant that is essentially an oil suspension with additional thickening agents and additives. When shear is applied, grease's initial high viscosity decreases, providing the impression of an oil-lubricated bearing with a density corresponding to the grease's base oil. Sometimes the term "grease" is used to describe lubricating substances, which are often soft solids or highly viscous liquids. However, these materials lack the shear thinning properties of conventional grease (thixotropic is the term for changing viscosity). The four forms of lubrication include boundary, mixed, elastohydrodynamic, and hydrodynamic regimes. Boundary lubrication is related to metal-to-metal contact between two sliding surfaces of the machine. When some equipment is first turned on or off, or when it is heavily loaded, the metal surface in a lubricated system may come into harsh contact. A wedge-shaped lubricant layer forms between the moving surface and the moving surface as border lubrication rapidly diminishes as sliding speed rises. As the possibility of asperity contact is reduced and layer thickness is increased, the coefficient of friction significantly decreases, creating a condition known as mixed lubrication. A complete layer of oil that supports and produces working clearance between the sliding surface is known as a hydrodynamic lubricant regime. High geometrical conformity between machine parts and minimal contact pressure between surfaces in relative motion are prerequisites for hydrodynamic lubrication.

Finally yet importantly, elastohydrodynamic lubrication is used when there is little conformity in the rolling motion between the moving elements and the contact zone. For instance, in a rolling element bearing, the race curve and the roller have significant differences. Due to the roller and inner race's oppositely curving shapes, the contact area was small (single point contact).

1.2 Problem Statement

Lubricating grease is typically necessary for machines and motors to function properly. A machine that is not properly lubricated could experience catastrophic failure due to increased grease deterioration. In order to keep the machine from breaking down while it is running, grease conditions must be closely maintained to prevent degradation. Due to mechanical work, high revolutions, or reduced consistency, the grease may have lost some of the oil that made up its makeup. It may also have degraded due to high temperatures or mixing with unsuitable grease. In addition, it's crucial to check the grease during maintenance procedures to make sure it hasn't been polluted by outside elements like water, dust, or worn metal. In a working environment, the lubricating grease also deteriorates with temperature, pressure, and load. A study of grease performance and degradation study is crucial in order to understand the grease characteristics at certain operating condition and to avoid damage to the lubricated components.

1.3 Research Objective

The hypothesis of this study is to study the tribology performance of Cu nanoparticle grease and MoS_2 microparticle grease that been formulate either it compatible to the commercial grease such as SKF LGGB 2/0.4 Bearing Grease and BIOGREASE Food Grade Grease Marlin-9. Therefore the objective of this research are:

i. To investigate the grease degradation in elastohydrodynamic lubrication
ii. To characterize the surface properties subjected to grease lubricant
iii. To evaluate the performance of palm oil based grease with additive and non additive grease

1.4 Scope of Project

The goals of this study are to evaluate the performance of four different types of grease: Marlin-9 Food Grade grease, SKF LGGB Biodegradable grease, Molybdenum Disulfide microparticle additive, and Copper nanoparticle additive. A Ducom four-ball tribology tester was used for the experiment the friction coefficient was examined. Using the Tabletop TM-1000, the wear scar diameter was measured. The three revolving ball samples were examined using a scanning electron microscope (SEM) to determine the wear scar morphology, and the composition of a particular location of the scar was plotted using EDX. In addition, Alicona IFM was used to analyze the profile form and surface roughness of the. Last but not least, TGA was utilized to analyze the mass loss and thermal stability of used grease containing Cu nanoparticles and MoS_2 microparticles.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction of Lubricants

In order to reduce friction and wear, lubrication involves introducing lubricant into the contact between loaded rolling or sliding bodies. Its use is to lessen interference between two surfaces that are in contact. There are four types of lubricants: oil, grease, penetrating lubricant, and dry lubricant. Oil and grease type lubricants are the most often used in daily application.

Long polymer chains make up oil lubricant, which also contains extra chemicals like detergents to stop deposits from forming, corrosion inhibitors to stop corrosion, and antioxidants to help it stay from oxidizing. When lubrication is required but resistance is not encountered, as well as when reassembling the machine is not possible, oil lubricant is utilized. Oil lubrication is not the greatest choice in a number of circumstances, including when the surface is moist because the oil will wash away because oil and water cannot interact. Oil cannot be added to the unclean or dusty portion in any other way. This is because applying oil to a soiled surface will make it more slick.

Combining oil with thickeners like lithium-based soaps produces grease lubricant. It can mix well with the lubricant in the oil and give it stickiness while acting as a barrier to shield surfaces from impurities that could damage them. When a machine includes fast-moving or delicate parts, grease is not the greatest choice for lubrication since its semisolid texture will slow it down and add too much resistance. In addition, the area is challenging to maintain clean since grease is flung around by fast-moving parts. This kind of lubricant is appropriate for use in chains, links, gears, and bearings. When we wish to lessen the frequency of the lubricating process, grease type is also used as the lubricant.

The penetrating lubricant comes next, acting as a shield for various stuck-bolt warriors. Penetrating lubricants, on the other hand, are not intended for long-term lubrication of these covered parts. These lubricant are made specifically to penetrate the surface's tiny fissures, enhance lubrication, and disintegrate rust because of their low viscosity. These lubricants can be used to free seized nuts or bolts. Remember that these lubricants are short-lived and should never be used on bearings or in place of other lubricants because they could harm the machine.

The last but certainly not least is dry lubricant, which has molecules that are slick and so reduce friction between surfaces. Dry lubricant is appropriate for use on hinges, locks, and other small parts that cannot become clogged with grease. It is also appropriate for surfaces that are exposed to extremely high temperatures or for materials that will begin to oxidize if an oil lubricant is employed. Dry lubricant is not appropriate in this situation since these types of lubricants will wash off if the surface is exposed to liquid or solvent.

2.2 Lubrication Types

Boundary, mixed, and full film lubrication are three different types of lubrication. To prevent wear, each type relies on a lubricant and an additive found in their composition. The first is boundary lubricant, which is used in situations with plenty of starts and stops as well as shock loading. In order to protect the surface in the event that the full film cannot be created owing to speed load or other circumstances, certain lubricants incorporate anti-wear (AW) or extreme pressure (EP) additives. To shield the metal from wear, these chemicals adhere to the surface of the metal and form a sacrificial layer. When two surfaces are in contact and only the EP or AW layer is shielding them, boundary lubrication takes place. This is not ideal since it results in excessive heat, friction, and other negative effects.

The next type of lubricant is a hybrid of hydrodynamic and boundary lubrication. The asperities still make contact with one another despite a lubricating layer separating the majority of the surface. The additive is used once more in this situation. It should be simpler to define lubrication if this process is better understood.

The whole film lubrication is the last type and can be divided into two types: hydrodynamic and elastohydrodynamic. When a fluid layer completely separates two surfaces during a sliding motion, hydrodynamic lubrication takes place. Similar to hydrodynamic lubrication, elastohydrodynamic lubrication takes place when the surface is rolling. In addition, the hydrodynamic lubricant is much thicker than the film layer, although the pressure on the film is higher. Elastohydrodynamic means that the film lubricates the rolling surface by elastically deforming it. Unevenness exists even on the most smooth and polished surface. They protrude from the surface, creating miniature peaks and valleys. These peaks are referred to as asperities. The lubricating film needs to be thicker than the asperities' length in order for the full film condition to be satisfied. The most efficient and sought-after form of lubrication, it safeguards the surface.

2.3 Grease Degradation

In general, we know that grease breaks down due to three main causes: physical, mechanical and chemicals. Rezasoltani and Khonsari 2019 (Rezasoltani & Khonsari, 2019) conducted an experiment to look at how lubricating grease degrades chemically. To track the chemical deterioration over an induced period of time, they employed two lithium complex grease in the experiment. In addition, the activation energies of the grease have also been measured using a Pressure Differential Scanning Calorimeter (PDSC). A heating chamber, a temperature controller, and an energy meter make up the experimental setup for heating grease samples. At the various test temperatures, this system evaluates pure energy absorption during chemical degradation. The energy absorption of the grease approached an identical maximum value in a shorter amount of time at high temperature and a longer amount of time at low temperature, according to the results. Two samples of chemically deteriorated grease were measured and compared for their ability to lubricate using a roller tester rig in order to validate the outcome. A mechanical degradation is also a type of physical degradation. It happens as a result of the lubricant being sheared. In a different paper that was published, Rezasoltani & Khonsari looked into mechanical deterioration as well. They looked at how grease degraded mechanically in the EHL line contact between two steel rollers. Rollers that are entirely coated in grease, slippage and grease separation, and the formation of liquid lubricant reservoirs are three separate lubrication phases that are observed and described. They provide the traction results for a grease that has gone through a protracted mechanical deterioration process. Not to mention, Hurley et al. (Hurley et al., 1998) used infrared spectroscopy to assess the chemistry-related change brought on by a straightforward thermal aging test on two lithium hydroxy stearate grease. The ability of the aged grease to lubricate a rolling contact under starvation conditions was assessed by measuring film thickness and oil release in that contact. The oxidation process is accelerated at 120 °C, according to infrared analysis, producing carboxylic acids and related species. The study also discovers that aged grease releases less oil since it has a thinner equilibrium coating.

2.3.1 Mechanical Degradation

Mechanical degradation deterioration in an elastohydrodynamic lubricant (EHL) line contact between two steel rollers is investigated by Rezasoltani & Khonsari (Rezasoltani & Khonsari, 2017). They observed and characterized three lubricating phases that is fully grease covered rollers, slippage and grease separation and formation of liquid lubricant reservoirs. For the grease that undergoing long-term mechanical degradation a traction result are present in the paper. A prediction model for assessment of reduction of grease consistency within EHL contact shown that both high shear rate and shear stress cause significant rapid mechanical degradation. It is demonstrated that grease within the contact area degrades rapidly (in a matter of minutes) and solid grease walls at the contact's sides seal this deteriorated grease. As long as the sealing effect of the walls exists, the severely deteriorated grease continues to lubricate the contact. The grease walls on the side of the EHL contact are likewise subjected to the mechanical life prediction model. It has been demonstrated that the sealing effect of walls may continue for a long time (several years).

2.3.2 Chemical Degradation

Chemical degradation of lubricating has been investigated by Rezasoltani & Khonsari (Rezasoltani & Khonsari, 2019a). They measure the activation energies and the chemical degradation of two lithium complex grease. Pressure Differential Scanning Calorimeter (PDSC) was calibrated according to ASTM D5483 standard and they performed the test followed the recommended procedure. Procedure using PDSC was started by weighed the sample in a small standard aluminum pan and then oxidized under 3.5 MPa of oxygen pressure at a constant test temperature. The heat flow difference was recorded and plotted as a function of time to measure the oxidation time. For the activation energy of the samples was calculated using the measured induction times at two different temperatures. The summary of the analysis show that the grease A has higher activation energy that grease B but the induction time of grease B is longer which is conclude that grease B more resistant to high temperature.

Beside that, formula for elastohydrodynamic lubricant that degraded chemically has been proposed by Kudish & Airapetyan (Kudish & Airapetyan, 2002). The new formula proposed takes into account stress induced degradation of polymer additive to lubricant on the basis of kinetic equation for polymer degradation. When a polymer additive degrades, it loses its viscosity irreversible resulting in a thinner lubricating coating and changes in other contact characterisrics therefore Kudish & Airapetyan proposed new formula to solve it numerically. The numerical and experimental result was compared and a very good agreement has been achieved that show the kinetic equaton can be used in further modelling.

2.4 Copper (Cu) Additive

Qiang et al. have investigated the tribological characteristics of a lithium-based grease with a nanometer-sized Cu component. They chose 1711 lithium-based greases with varying concentrations of copper nanoparticles (0.15, 0.25, 0.35, and 0.45 percent) as their research subjects. Their research reveals that nanoparticles have an average diameter of 10 nm and a spherical diameter. Using transmission electron microscopy (TEM, morphology results demonstrate that Cu nanoparticles have a very narrow size distribution, are evenly disseminated without condensing, and were obtained. According to tests by Qiang et al., 0.25 %w/w of nanoscale Cu is the ideal additive concentration in 1171 lithium-based grease. The grease's inability to function as a lubricant when added in excess amounts of nanometers of copper results in an increase in friction coefficient and wear scar diameter (Qiang et al., 2017).

Additionally, Nan et al. looked at how Cu nanoparticles affected the tribological characteristics of attapulgite grease. The chemical formula of attapulgite, a nanoscale phyllosilicate mineral, is $(\mathrm{Mg,Al,Fe})_5\mathrm{Si}_8\mathrm{O}_{20}(\mathrm{OH}_2)_4$. This grease is an eco-friendly and green lubricant. The grease with two weight percent Cu nanoparticles has the best tribological properties, according to Nan et al. The initial result reveals that the friction coefficient, which is less than the base grease, was 0.11 after approximately 5 minutes of operation, then it increased to almost 0.12 and stayed stable during the remainder time. The study also looked at how temperature and applied load affected the lubricating grease's friction coefficient. Cu nanoparticles could enhance the basic grease's anti-wear and friction reduction capabilities for all three test temperature ranges (30, 100, and 200 °C). Due to the presence of Cu nanoparticles, base grease's coefficient of friction dropped simultaneously when loads of 50, 100, and 200 Newton were applied. According to studies, Cu nanoparticles can enhance the tribological properties of grease based on attapulgite, and their ideal additive level is two weight percent (Nan et al., 2015).

Cu nanoparticles were previously researched in an oil lubricant by Kuznetsov et al in 1990. Their investigation concentrated on how surface-modified Cu nanoparticles as a 50CC oil additive affected wear and friction parameters. Because the real service temperature of lubricating oil is relatively high—around 90–100 °C—it is crucial to investigate how temperature affects the lubricating characteristics of Cu nanoparticles as additions. According to their research, oil containing Cu nanoparticles has a lower wear scar diameter and friction coefficient than pure oil. In conclusion, Cu nanoparticles have improved tribological characteristics at higher temperatures. The worn ball's morphologies also demonstrate a favorable outcome: the ball lubricated with oil containing Cu nanoparticles is significantly smoother and exhibits less severe scuffing (Kuznetsov et al., 1990).

2.5 Molybdenum Disulfide (MoS_2) Additive

Using a block-on-ring test, the lubricating characteristics of MoS_2 and CaF_2 components with comparable size as oil additives were investigated. According to the results of the experimental inquiry, 1-1.5 weight percent was the ideal ratio for the MoS_2 and CaF_2 additive. Additionally, Bas et al. discovered that for MoS_2 and CaF_2, respectively, the additive can lower coefficient friction by about 50% and 37.5 percent (Baş et al., 2022). Additionally, MoS_2

outperforms CaF_2 in the surface roughness test results. Using a scanning electron microscope, the morphological pictures of the worn surface and additive were studied, revealing numerous pits and plowing on the surface. The oil was unable to penetrate some sections of the sample surface, resulting in the formation of black patches on the surface. Finally, even under working circumstances in a room, MoS_2 and CaF_2 additives have a lowering effect on friction.

In addition, Liu et al. investigated the lubricating mechanism of MoS_2 and NiTi filled in surface microcavities as well as the tribological performance of material M, MM, and MMN in a broad load range. The M, MM, and MMN samples are simplified variations of the sample's M50, M50- MoS_2, and M50-(MoS_2-NiTi) compositions, respectively. The researcher discovered that, particularly in high-load applications, the tribological performance of MM and MMN was superior than M. This is as a result of the additive. When compared to MM and MMN that have additive, MM has the worst friction coefficient and wear rate. When the load is 16–21 N, the friction coefficient of MMN is lowered by 35%, and the wear rate of MMN is roughly 30% lower than MM (Liu et al., 2021). According to the morphological analysis, MMN only contains minor debris and peeling pits on the work surface. As the load is increased, the worn surface becomes smooth and reveals a dark black area. On the 21N, the surface roughness of wear and the coefficient of friction both decrease but are inconsistent. The outcome of the wear morphology reveals that under specific circumstances, the current additive can lower the friction coefficient and surface roughness.

The performance of MoS_2 under the application of a solid lubricating coating was also examined by Huang et al. They used pin-on-disc testing to examine how the MoS_2 thin film and DLC solid coating behaved when sliding across air under various test settings. In addition, the combined properties of a solid thin film and a liquid lubricant oil were investigated. Results from

Huang et al. demonstrate that grease lubrication reduces the influence of liquid lubricant on the friction of MoS_2 film and DLC coating film by more than oil lubrication. Under various lubrication conditions, the wear resistance properties of magnetron sputtered MoS_2 film and chemical vapor deposited DLC coating display diverse effects. The experiment's findings indicate that although boosting the wear resistance of DLC film, liquid lubrication significantly reduced the wearability of the MoS_2 coating (Huang et al., 2010). In conclusion, although it was the opposite of the DLC, the average specific wear rate of MoS_2 film lubricated with grease was lower than that lubricated with oil.

CHAPTER 3

METHODOLOGY

3.1 Overview

The lubricant of this project was introduced in this chapter. In addition, the fundamental details and uses of each machine were explained along with the experiment's approach. The equipment employed included a four-ball tester, Hitachi tabletop microscopy, a scanning electron microscope (SEM), an Alicona Infinite Focus Microscope (IFM), and a Thermogalvanomatric analysis (TGA). The machine provides results in the form of friction coefficient, wear scar diameter, wear morphology, element composition, surface roughness, and lubricant mass loss.

3.2 Lubricants

Four different types of grease were tested in this study; two of them were palm oil-based grease with additive that were produced in a lab by master students, and the other two were commercial mineral-based grease available in the market. The palm oil grease were formulated with palm olein and lithium stearate thickener to produce grease semi-solid structure. The grease was then added with molybdenum disulfide microparticles and the other one with copper nanoparticles additives. For comparison, the mineral based BIOGREASE Food Grade Grease Marlin-9 and SKF LGGB 2/0.4 Bearing Grease were tested.

Figure 3.1 The grease tested in this study (a) Palm oil grease with Cu nanoparticle additive (b) Palm oil grease with MoS_2 microparticle additive (c) SKF LGGB Biodegradable grease amd (d) Marlin-9 Food Grade Grease

3.3 Four Ball Tester

This machine has three variables that are controllable: the applied weight, the rotational speed, and the temperature of the ball cup. The device is used to assess the lubricant's friction and wear properties under various test conditions. The machine operates by pressing three balls that are held together at the bottom against a ball that is revolving at the top and submerged in a test lubricant. While the three lower balls are kept together in the ball cup, the revolving top ball is assisted by a collect that is coupled to the machine's vertical rotating shaft. Figure 3.2 epicts the configuration of the ball bearing for four ball tester machine (Aiman & Syahrullail, 2017). A steel ball is rotated against three stationary, lubricated steel balls during the 4-Ball Wear test at a specific load, speed, temperature, and time as per ASTM D2266 (grease) or ASTM D-4172 (oil). The wear scar on the three stationary balls will be smaller the better the lubricant performs in preventing wear. The three-wear scars are measured and the average is published when the test is complete. Throughout the 60-minute test, the coefficient of friction is also measured, and the average is presented at the conclusion. Since grease was the material employed in this investigation, the

ASTM D2266 Standard was used to examine the wear scar shape and coefficient of friction. In addition, the relationship between high temperature (100°C) and high load (100kg @ 981 N) also been studied. Table 3.1 displays the variables chosen for the various conditions.

Table 3.1 Variable for different test used in this study

Condition	Temperature	Load	Time	Speed
1	75℃	40kg	60 min	1200rpm
2	100℃	40kg	60 min	1200rpm
3	75℃	100kg	60 min	1200rpm

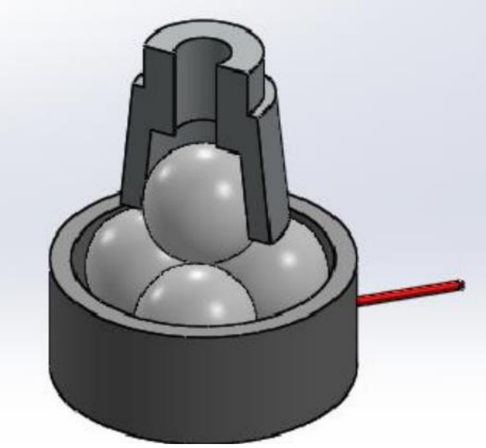


Figure 3.2 Schematic diagram of four ball tester. (Aiman & Syahrullail, 2017)

Figure 3.3 DUCOM Four-Ball Tester Machine

3.4 Tabletop Microscopy (TM1000)

Tabletop scanning electron microscopy is a reliable and simple tool for visualizing surface attributes at a higher resolution and deeper focus depth than is possible with light microscopy. Back scattered electrons produced by a 15 keV electron beam are used to create the image.

By scanning the surface of samples with a focussed electron beam, scanning electron microscopes create images of the samples. The microscope's top produces the beam electrons by heating a metallic filament. Through a succession of electromagnetic lenses, the electron beam travels vertically before being focused and pointed towards the sample. In response to the electron's interactions with the sample's atoms, secondary electrons are ejected from the sample; these electrons are then transformed by a detector into signals that reveal the sample's surface topography and composition. Within a compartment that is vacuumed, the specimens are examined.

Figure 3.4 Hitachi TM1000-Tabletop Microscope machine

3.5 Scanning Electron Microscope (SEM)

In theory, the scanning electron microscope allows us to see material by moving a tiny electron beam over a sample's surface in time with the cathode ray tube's display spot (CRT). A tungsten cathode or a field emission can both emit electrons. The brightness of the CRT spot is regulated by an amplified version of the detected signal, and a detector measures the strength of the secondary signal coming from the specimen. A high-performance, user-friendly scanning electron microscope with new upgrades that provide the greatest outcomes for a variety of applications is the Hitachi S-3400N SEM. It contains a completely eccentric 5-axis motorized stage that can accommodate samples up to 25 cm in diameter, along with both Secondary (SE) and Backscatter (BSE) Electron Detectors. In low vacuum mode, the instrument is fitted with a Deben Peltier Coolstage to reduce moisture loss, and the Variable Pressure mode permits BSE observation from 6-270Pa. This device can magnify objects up to 300 000 times.

Figure 3.5 Hitachi S-3400N SEM

3.6 Alicona Infinite Focus Microscope (IFM)

A 3D surface measurement optical tool is the Alicona Infinite Focus (IFM). Its working principle uses vertical scanning in conjunction with an optical system's shallow depth of focus to extract topographical and color information from the focus variation. This is reconstructed using novel and distinctive techniques into a single 3D data set with precise topographical data. The instrument is suited for the surface investigation of both homogeneous and complex materials because "Z" resolution can be as low as 10nm. Area, volume, and roughness measurements can be obtained using this device at 2.5x–100x magnification.

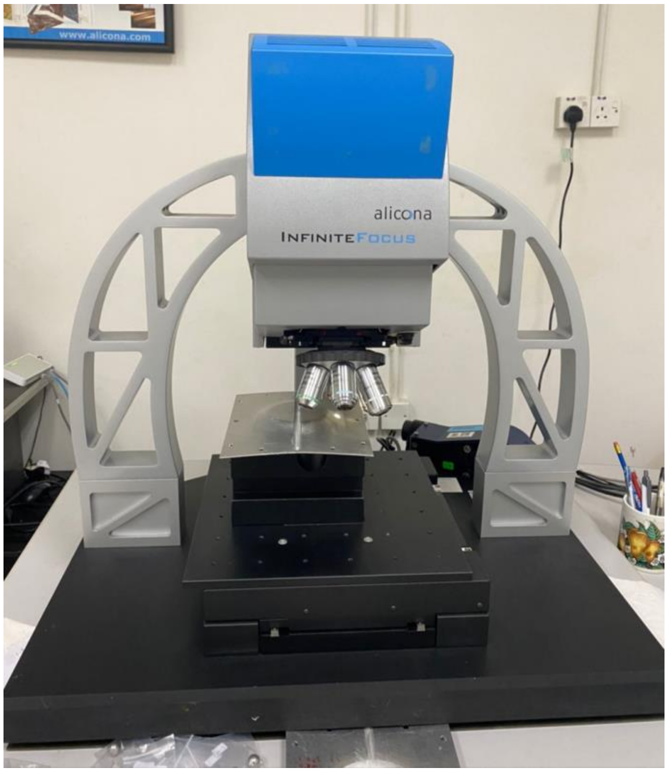


Figure 3.6 Alicona IFM-Optical 3D surface metrology

3.7 Thermogravimetric Analysis

The physical and chemical properties of materials are measured as a function of changing temperature (with a constant heating rate) or as a function of time using the thermal gravimetric analysis (TGA) technique (with constant temperature or constant mass loss). The second order phase transition, which includes vaporization, sublimation, absorption and desorption or chemisorption, desolations, and others, can be studied using TGA (especially dehydration and decomposition). TGA is frequently used to identify certain properties of materials that show either mass loss or gain owing to oxidation, breakdown, or loss of volatile substances like moisture. TGA is frequently used for the following tasks: (1) characterizing materials through examination of their distinctive decomposition patterns; (2) researching degradation mechanisms and reaction kinetics; (3) figuring out how much organic and inorganic material is present in a sample; and (4) performing chemical analyses. Temperature is plotted on the x-axis of a TGA curve, and mass loss is plotted on the y-axis (or time and most of the time a direct heating rate). In this study, the mass loss of waste grease were analysed and it thermal stability can be obtained by the TGA curve graph.

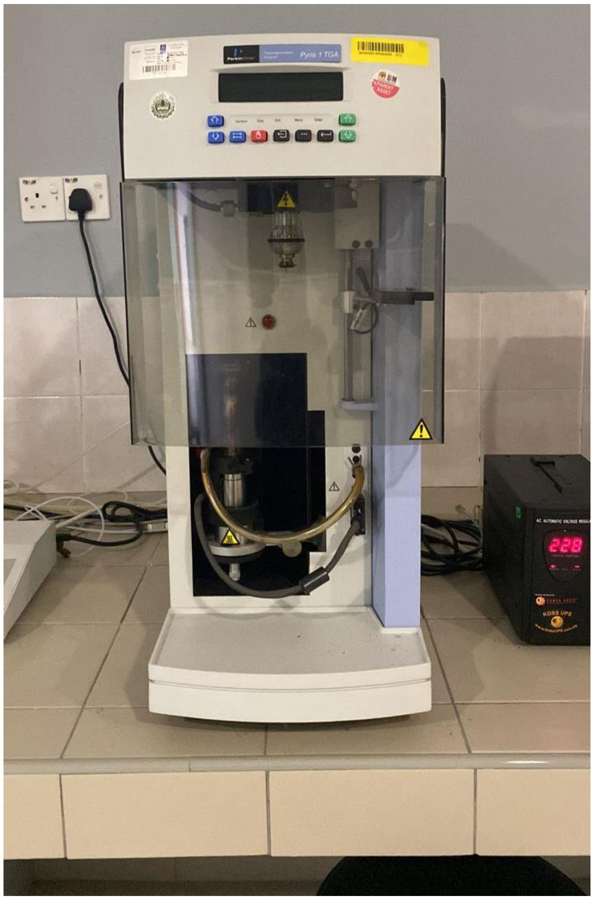


Figure 3.7 Thermogravimetric analyzer

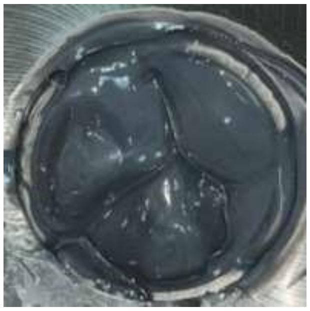
CHAPTER 4

RESULT AND DISCUSSION

4.1 Physical Degradation

Referring to Table 4.1, the condition of grease after the experiment can be observed by comparing the grease condition after three different test. For Cu additive, it can be observed that the colour condition of grease turn from yellowish to brown colour under ASTM D2266 Standard to high load test respectively. For MoS_2 additive, there no obvious observation can be recorded during after the experiment since the colour of the grease is a dark grey. As a comparison for the 2 additives, the waste is keep for several days as an observation. During the observation, there Cu tend to oxidized faster compared to MoS_2 because the colour of Cu grease turn to blue for all type of test. Besides that for the both additive grease there only small increasing in temperature around 2-3 °Celcius. For LGGB and Food Grade, the grease around the contact show a burn and scorched condition. For LGGB, the grease under the high load test show it the worst with a very dark sign of scorched. During the experiment, the sign of increasing temperature also severe same as additive grease which is only up to 3 °Celcius. Last but not least, is Food Grade Grease where at the high temperature the colour of the grease changing from a clear to a rust brown around the ball contact. It become more severe at high load test, where there a the rust brown colour spread widely. Beside that, during the experiment, high increasing in temperature can be observed that almost up to 10 degree.

Table 4.1 Condition of grease after the experiment.

Grease	ASTM D2266	High Temperature	High Load
Cu			
MoS_2			
LGGB			
Food Grade			

4.2 Mechanical Degradation

4.2.1 Coefficient of Friction

In this study, the effect of load and temperature on the tribological performance of palm-oil-based grease were investigated using a four-ball tester according to the standard test of ASTM D2266. Tests were conducted with 40kg and 100 kg load and 75℃ and 100℃ temperature. The other parameters were constant such as rotational speed is 1200rpm and test duration was 60 minutes for all cases. The result focused on the wear scar diameter, friction coefficient and flash temperature. Following the completion of the wear test experiments, the ball wear condition and lubricant properties were observed. Figure 4.1, 4.2 and 4.3 show the friction coefficient graph of 4 types of greases (palm oil with Cu additive, palm oil with MoS_2 additive, Biodegradable Grease, Food Grade Grease) under a different type of testing condition which is following the ASTM D2266 Standard, high temperature application (approximately 100 ℃) and high load application (approximately 100 kg). While, Figure 4.5 and 4.6 is the comparisons of COF for Cu nano additive and MoS_2 micro additive tested in different condition. Average COF were presented in Figure 4.4.

The finding show that for ASTM D2266 Standard, Cu has the lowest coefficient of friction which is 0.023 followed by MoS_2 with 0.035. This result were expected to happened because Cu has the smaller particle (nano size) compare to MoS_2 (micro size). LGGB has the higher COF compare to Cu and MoS_2 because after 2500 second the COF were increasing from 0.04 to 0.06 and it reduce to 0.04 again before reaching 3000 second. It is different for Cu and MoS_2 that has the start up COF at 0.04 but slowly reduce around 500 second for Cu and around 200 second for MoS_2. The graph show, MoS_2 keep stable till the end of the experiment, while there some fluctuation at the end for Cu nanoparticle. Food Grade show the highest COF for all test and all it COF graph show unstable result with many fluctuation.

After that, the effect of temperature were studied and show MoS_2 has the lowest COF compared to other. The main reason for MoS_2 has the lowest COF can be seen from Figure 4.2. Based on the figure, the start up COF around 0.03 and increased to 0.04 but start reduce at the time 500 second. It reduce until it reach around 0.01 COF. The COF of Cu has a small different compare to LGGB COF in high temperature test. It can be seen in the Figure 4.2 that the these two grease has overlapping line but, LGGB has two obvious peak that reach 0.11 and 0.08 COF. This is the main result why these two has almost similar COF but LGGB has higher value compare to Cu.

After that, the experiment were continue but the load were change from 40kg to 70kg. The result show that Cu still has the lowest COF followed by MoS_2, LGGB and Food Grade. But the average COF for the first three grease show a slightly different only to LGGB. This can be proved, by looking at the Figure 4.3 where the graph of Cu, MoS_2 and LGGB were overlapping to each other. Beside that, MoS_2 a show it has less fluctuate compare to Cu and MoS_2. What can be conclude by the smoothest line of MoS_2 is, this grease is most stable compare to other and the agglomeration is lowest compare to Cu. During the experiment, the flash temperature were observed for all type of grease. MoS_2 and Cu running time temperature has 4 degree increasing only. LGGB and Food grade faced flash temperature where LGGB increased to 88 °Celcius and Food Grade increase to around 98 °Celcius from 75 °Celcius during high load test.