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THE EFFECT OF TUNGSTEN DISULFIDE ADDITIVES AND SODIUM DODECYL SULFATE ON BASE OILS COMBINATIONS COMPARED TO LUBRICANTS 10W-40 TO REDUCE FRICTION

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ABSTRACT

The research was conducted to evaluate the effect of Tungsten Disulfide (WS_2)as a friction modifierandSodium Dodecyl Sulfate additives agglomeration of WS_2 particles in the lubricant. To get the maximum composition, 13 samples were tested with composition variation, and the maximum composition obtained was of 0.1% and 0.2% of Nano WS_2 and SDS respectively. The analysis was performed by using 2 test instruments such as Four-Ball Scar Diameter (FBSD) and High-Frequency Reciprocating Rig (HFRR). FBSD value without WS_2 Nano additive was 0.27mm while there was a decrease in scar diameter by 11% to 0.22mm with WS_2 addition. Meanwhile, HFRR wears scar analysis accounted for a value of 183.5 µm without WS_2 Nano additive and there was a decrease of 40.8% to 108.5 µm with the addition. Next, there was a decrease of friction coefficient after the addition of WS_2 Nano additives by 7.62%, which was from 0.118 (without Nano additive) to 0.109, in the friction test using HFRR instrument. From these results, the use of WS_2 and SDS as additives is able to reduce the physical characteristics based on the friction and scar diameter during the mechanical interactions.

Keywords: Tungsten disulfide (WS_2), Sodium Dodecyl Sulfate (SDS), SAE 10W-40 Lubricant, Friction Coefficient, Scar Diameter

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INTRODUCTION

When a machine is working, there are interactions or mechanic contact between each and every element. This causes the elements to rub against each other and lead to the machine being worn-out. Friction is defined as the energy dissipated between surfaces caused by uneven material surface[1,2]. It affects energy efficiency, machine mechanical endurance and wears and tear. Repeated uncontrollable friction results in wear and tear which damages material surfaces due to dislocation[3]. To overcome this matter, the lubricant is used to reduce friction as it provides a second layer which lowered the burden. Besides that, the application of Nanotechnology on lubricant has become one of the solutions to reduce friction.

Tungsten Disulfide (WS_2) is a Nanoparticle additive used in this study. WS_2 Nanoparticles contain layered structured with W elements located between the layers of S elements. The configuration allows WS_2 to have the characteristics that enable it to react with metal substrate and creates extreme pressure (EP) additives at high temperature and pressure. EP additives contain a big number of S elements, which tend to sacrifice the element in layers of film. Therefore, the element is suitable in the application at high load and pressure[11]. The S element is very good at lubrication, and it starts the reaction to form any corrosive effect[3]. WS_2 has high thermal stability and strong bonds between layers. As a result, the reactivity is preventable and low. The addition of Nanoparticles in the lubricant enables them to penetrate into various uneven areas and filled the gap between them. Eventually, film layers able to endure high

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pressure will be formed. Therefore, Nanoparticles need to have smaller or at least similar sizes to the hardness of material they are in contact with in order to penetrate and fill the pores efficiently[4,5,6,7,8]. The presence of Nano additives in lubricant makes an energyto be used better and more efficiently compared to conventional lubricant or the one without Nano additive as it has the protection against components' wear and tears. The asperities of material surfaces build up the pressure and temperature which helps in S element sacrifices[12].WS_2 has a high potential to become the solution to embrittlement (the brittle and easy to break characteristic due to hydrogen diffusion in metal) because it has the ability to form tribofilm on steel substrate which efficiently prevents hydrogen diffusion[13].

The usage of Nanoparticles as an additive has been analyzed by scientists; nevertheless, the number of issues needs to be handled like thermodynamic instability. Lubricant layered by inorganic matters is bonded weakly by Van der Waals interaction like WS_2 which works by physico-adsorption (physical absorption) to substrate and is moved by sliding with a very low friction coefficient[4,14,15,16]. WS_2has the ability as a wonderful friction modifier[17,18] despite a little problem on the stability of its particles dispersion[19,20,21,22]. Nanoparticles colloid and the compatibility with the base oil or the additives[4,5,6]while the study about WS_2 and SDS Nanotechnology application in lubricants has been done by several scientists[27,28,29]. In order to be applied effectively, WS_2 Nanoparticles need to be stable in the colloid form so that when the particles are at base oil, its tribology properties go well without disturbing any other additives in the lubricant[23,24]. Therefore, the usage of surfactant as the dispersion agent can maintain WS_2Nanoparticles in the colloid phase to be stable for a long time. As long as the surfactant consists of amphiphilic substance, SDS is one of the examples;the polar surface of the oil is protected.

In this study, 10W-40 lubricant is used. It is one of the automotive lubricants which used generally by people. To know the effect of WS_2Nanoparticles in the lubricant, a laboratory scale study based on American Standard Testing and Measurement (ASTM) is conducted. Reduction of friction, wear and tear and the stability of WS_2Nanoparticles by using surfactant in the lubricant are tested and analyzed. The results of this study are expected to give a descriptive explanation about effective and efficient Nanoparticles additives which later can be applied as an innovative to lubricant in the future.

EXPERIMENTAL

Material and Methods

Formulation of Optimum Base Oil and Additives (WS_2 and SDS)

WS_2Nanoparticles used in the study was bought at MK Impex Corp with the characteristic of 99.9% purity, 0.6-micron APS and a heat resistance of -450^{0}C to 1200^{0}C at normal or vacuum pressure. SDS surfactant used was bought from Sigma Aldrich with the characteristic of 288.38 g mol^{-1} of molar mass, 1.01g/cm^3 of density and melting point at 206^{0}C.Base oil type HVI 95 and HVI 60 used as base oil group 1 is produced by Pertamina Indonesia with characteristic of <90% saturation, >0.03% of sulfur content, viscosity index between 80-120, Solvent-Refined based production, stable at a temperature between 32^{0}F – 150^{0}F. Whereas Yubase 8 as base oil group 2 is produced by SK Energy Korea with >90% saturation, <0.03% sulfur content, viscosity index above 120 and hydrocracking based production.

The process of the formulation was performed by blending method within 1 hour with 50^{o}C stirred for 330 rpm. Firstly, the base oils which were Yubase 8, HVI-95 and HVI-60 were prepared to be added by the additives with different compositions shown by Table-1. The blending process involved SDS was conducted to prevent the presence of agglomerations, formed by the WS_2. The control variables were the base oils which were not added by each additive.

After the mixing process was completed, the mixture was directly being tested its friction characteristic and protection against wear and tear with Four Ball machine based on America Society for Testing and Materials (ASTM D 4172) and HFRR (ASTM D 6079) to get the description for the optimum composition of WS_2 and surfactant.

During the process of blending with the base oils, the additives of WS_2 tend to form agglomeration. This condition also leads to the formation of precipitation which is inappropriate, and it can cause rough surface leading to high coefficient of frictions. There are two factors caused this condition, first and foremost is due to the undistributed amount of particles on the surface, while the second is the temperature. Therefore, stirring and temperatures are essential in the blending process. The process of

blending of base oils and the additives were performed in stirring condition for one hour within 50°C and 330 rpm. The following Table-2 was the optimum concentration obtained based on the experimental process.
The optimum concentration obtained from each base oils then were applied to lubricants type SAE 10W-40. The blending process was performed by involving 100°C inside Fume Hood. Each sample was applied according to the previous formulation, and it is shown by following Table-2. After the blending process is completed, the samples were tested based on SAE J300 followed by Four-Ball and HFRR.

Table-1: The Formulation of Optimum Concentration between $WS_2$and SDS

Base Oil	Concentration		Code
	WS_2 (%w)	SDS (%w)	
Yubase 8	0.025	0.025	Y1
	0.05	0.05	Y2
	0.25	0.25	Y3
	0.025	0.05	Y4
	0.25	0.05	Y5
	0.1	0.2	Y6
HVI-95	0.025	0.025	H1
	0.05	0.05	H2
	0.25	0.25	H3
	0.025	0.05	H4
	0.25	0.05	H5
	0.1	0.2	H6
HVI-60	0.1	0.2	V1

Table-2: Optimum Concentration of each Base Oil

Base Oil	Concentration		Code
	WS_2 (%w)	SDS (%w)	
Yubase 8	0.1	0.2	Y6
HVI-95	0.1	0.2	H6
HVI-60	0.1	0.2	V1
10W-40 Lubricants	0.1	0.2	W1

The formulation of maximum concentration was conducted in order to fulfill SAE and API service. And, we found that the blending process was optimally involved with 0.1 (%w) of WS_2 and 0.1 (%w) of SDS Surfactant. In this study, the three base oils were combined by blending process which involves modifier to form lubricants. The process of blending theoretically has been determined, however, laboratory analysis showed different values regarding the concentrations.

Four-Ball Testing

The Four-Ball testing aims to analyses lubricant endurance to friction and wear and tear. The formulated lubricant was tested its characteristic based on ASTM D 4172 standard. Four-Ball test machine used was Stanhope Sete in which the ball is made of chromium alloy based on the material standard from American Iron and Steel Institute (AISI E-5200, with a diameter of 12.7 mm/0.5 inches), Extra Polish (EP) grade and Rockwell C hardness between 64 - 66. The result obtained from the test is the measure of scar in the test ball[30].

HFRR Test

The aim of the HFRR test is to measure friction coefficient, scar diameter and the characteristic of film lubrication. The test is conducted based on the requirement by the American Society for Testing and Materials (ASTM D 6079). The test ball has the specification as follow: made of steel AISI E-52100 grade 25 per ANSI B3.12, the diameter of 6.00 mm, Rockwell Hardness "C" scale (HRC) number 58 - 66 according to E 18 test and surface hardness less than 0.05 µm Ra. Disc test with the size of 10 mm made of steel AISI E-52100 having Vickers hardness level of "HV 30" based on E92 specification with a scale between 190 and 210, layered by surface asperity less than 0.02 µm Ra[31].

Physical Characterizations

The combination of three base oils must be analyzed based on its physical characterizations. In this study, there are 5 physical properties analyzed to obtain the standard grade of the 10W-40 lubricants. They are density, flash point, pour point, viscosity, cold cranking simulator, and copper strip corrosion. The analysis of first-four properties was performed according to ASTM standards which are shown in Table-1 afterward, while the Copper Strip Corrosion (CSC) was analyzed based on the ASTM D 130 methods. As the SDS surfactant dissolved with distilled water was used, this test was conducted to determine the water contents within the lubricant. Water content is a potential factor that can produce corrosion to the machine so that the wear could have occurred.

Results and Discussion

Characterization of Additives WS_2 Particles

In this study, the Nano additives WS_2 Particles are characterized by Transmission Electron Microscopy (TEM) to determine the Nano size. The particles image is shown in Fig.-1 below.

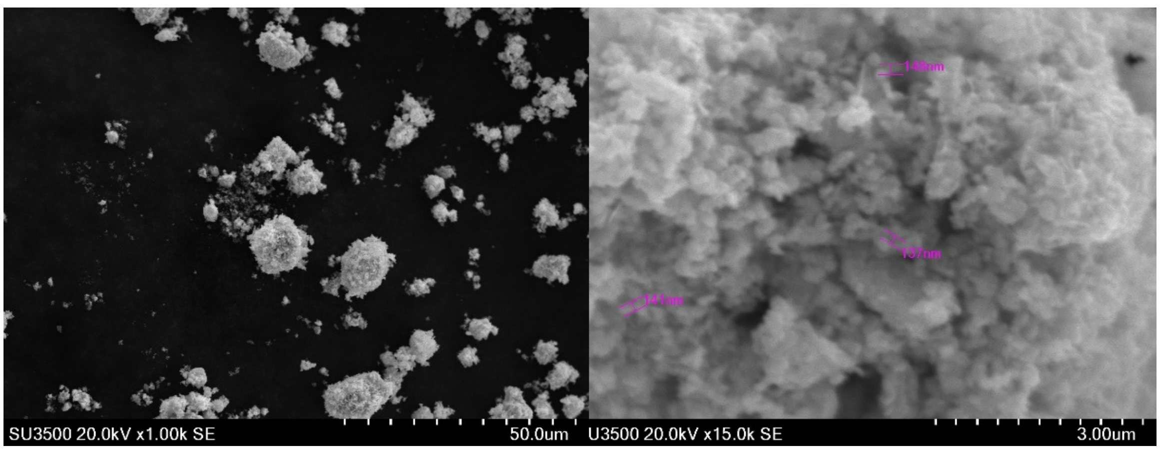

Fig.-1: The TEM Photographs of Tungsten Disulfide (WS_2)

Based on the TEM's test, the additives WS_2 particles used in this study were observed within 50.0 µm enhancement. Then, the test was enhanced to 3.00 µm to calculate the nanoscale, and the results showed that the particles were approximately 137 nm and 148 nm.

Four-Ball Test Result

Protection characteristic against wear and tear is indicated from the measure of scar diameter on the test ball using Four-Ball instrument. The C codes are the control variables, so that the effects of particle additives could be determined. The result of the test is shown in Table-3 below.

Table-3: The Average Wear Scar Diameter of Samples

Base Oil	Concentration		Code	Average Wear Scar Diameter (mm)
	WS_2 (%w)	SDS (%w)		
Yubase 8	-	-	C	0.47
	0.025	0.025	Y1	0.51
	0.05	0.05	Y2	0.38
	0.25	0.25	Y3	0.41
	0.025	0.05	Y4	0.4
	0.25	0.05	Y5	0.36
	0.1	0.2	Y6	0.29
HVI-95	-	-	C	0.73

	0.025	0.025	H1	0.42
	0.05	0.05	H2	0.54
	0.25	0.25	H3	0.43
	0.025	0.05	H4	0.6
	0.25	0.05	H5	0.5
	0.1	0.2	H6	0.33
HVI-60	-	-	C	0.72
	0.1	0.2	V1	0.31
10W-40	-	-	C	0.28
	0.1	0.2	W	0.25

Table-1 shows a decreasing trend to scar diameter for each sample. In Yubase 8 base lubricant samples, sample code Y1 has the biggest scar diameter of 0.51 whereas the smallest is in sample code Y6 of 0.29 of diameter. Meanwhile, HVI-95 base lubricants gave a fluctuated trend. Despite the trend, sample code H4 is the sample with the biggest scar diameter (0.6) and scar diameter got smaller to 0.33 as the smallest value.

Based on the four-ball tests results, every control samples of base oils had the highest average wear scar diameters, while the smaller concentrations of additives affected fluctuating values of scar gradually, such as samples C of Yubase 8 to Y1, Y2 and Y3. These conditions were based on the distribution of WS_2 and SDS as well as the stability of their composition during the process of blending. Consequently, the blending process of each base oils to form the lubricants were decided based on physical characteristics which agreed to the SAE and ASTM standards, which indicated by code W. The following Table-4 is the comparison between the physical properties of the samples obtained and the standard values based on ASTM standard values.

Table-4: Physical Characterizations of Lubricant based on a Combination of Three Base Oils Used

Characterizations	Units	Methods	Standard Values	Samples	
				C	W
Density	gr/cm^3	ASTM D 4052, 1250	0.7981-1.1607	0.8719	0.8722
Flash Point	^{o}C	ASTM D 92	>200	228	228
Pour Point	^{o}C	ASTM D 97	≤-33	-36	-36
Kinematic Viscosity @40^{o}C	cSt	ASTM D 445	36-340	96	97.62
Kinematic Viscosity @100^{o}C	cSt	ASTM D 445	12.5-16.3	14.12	14.15
Viscosity Index		ASTM D 2270	≥100	151	148
CCS Viscosity @-25^{o}C	Cp	ASTM D5293	≤7000	6648	4716

According to Table-1 previously, it can be seen that the sample which blended with additives matches the standard values of ASTM standards.Meanwhile, the CSC test showed no corrosion occurred on the copper strip, indicating the some of the distilled water that has been used in the blending process were not found. The next characterization was the Fourball test to determine the effect of additives in producing the scar diameter. The following Fig.-2 shows the results of the base oils and lubricants.

Figure-2 shows the comparison of each sample with their control samples. It can be seen from the bar chart, the samples which added by the particle additives had a decreasing value of scar diameter. The most significant decreasing values of scar-diameter were in HVI-95 and HVI-60, which accounted for up to 60%, whereas the Yubase 8 base oil was over 30% of compared to the control. Given that the previous assumption suggested the combination of three base oils could provide a small diameter, the percentage of declining in the diameter values was the lowest for 11%. Interestingly, although the value of sample 10W-40 was the lowest in diameter among all samples, the beginning value of scar diameter in control was the lowest, suggesting the combinations significantly occurred.

The Fourball tests involved within 75±2^{o}C for one hour with 40±0.2 Kgf of load force. Based on the results, it can be observed that the samples of every base oils without additives produced higher scar diameter than those with additives, indicating the additives provided resistance to the high temperature

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and prevented the oxidation process. This implies the protection of additives during the interaction events, so that the friction could be reduced, which is shown by the HFRR test.

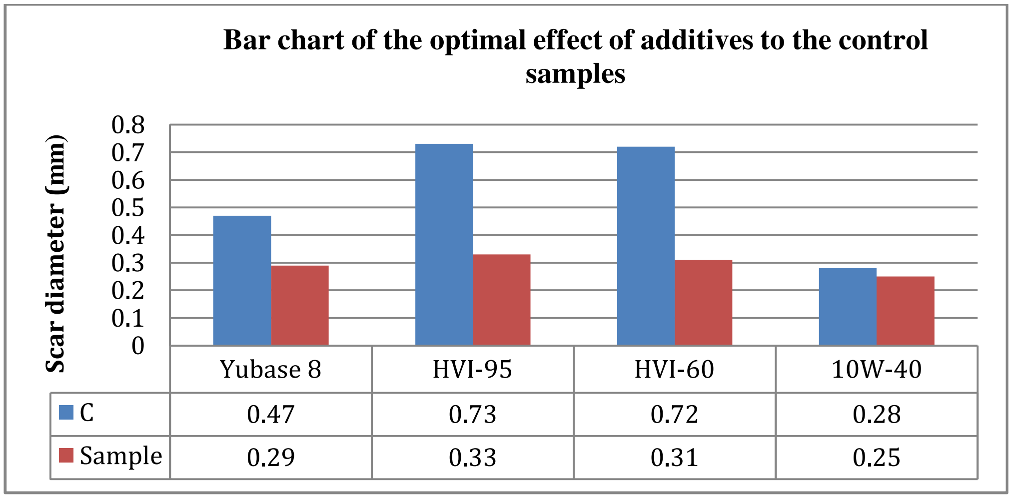
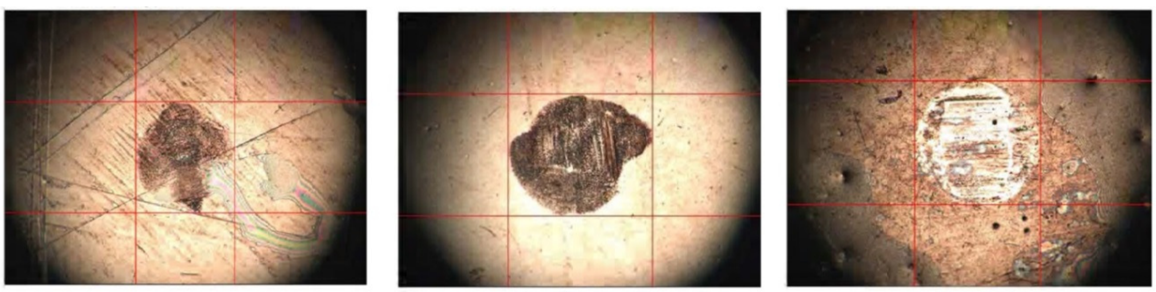


Fig.-2: The Results of Four-Ball Scar Diameter Test to Determine Optimum Concentration of WS_2 + SDS

HighFrequency Reciprocating Rig (HFRR) Test
Wear Scar



Sample Y6 Sample H6 Sample V1

Fig.-3: Microscopic Observations of HFRR Test

According to figure two above, it can be observed that the scar diameter resulted by Sample Y6 is the smallest among those three samples. In the meantime, in sample VI, the centered-surface is almost covered by the scar forming a circle-shaped. The diameter of the scar of the base oils samples was shown by the following Fig.-4.

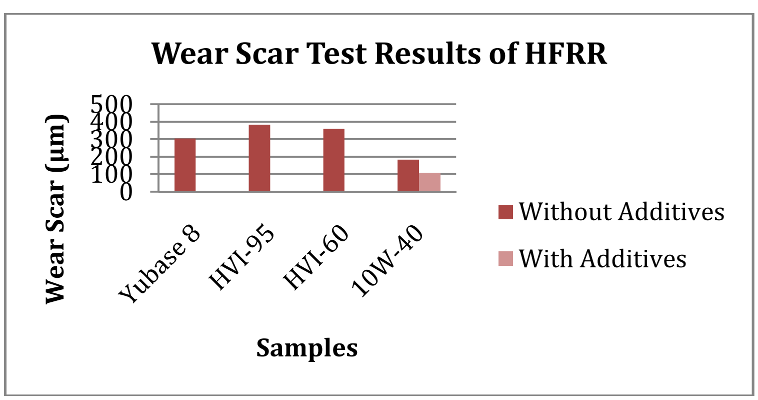


Fig.-4: Wear Scar Test Results of HFRR of Base Oils Compared to the Lubricants Obtained

The wear scar test of lubricants 10W-40 showed a significant difference. The percentage of wear scar was almost a third compared to the three base oils. The scar diameter of sample 10W-40 is displayed by Fig.-5 below.

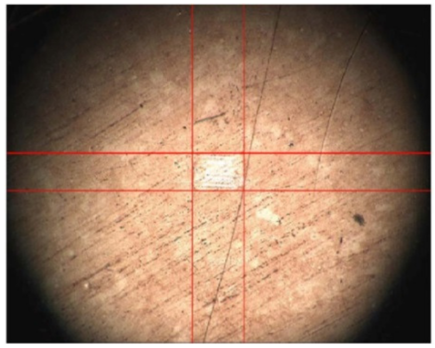


Fig.-5: HFRR test of Sample 10W-40 with Additives

Friction Coefficient

After the maximum of each composition from each tests using Four-Ball instrument is known, the test friction coefficient is only calculated for the best composition out of the instrument test. The sample was prepared to be tested, Fig.-6 below shows the test result.

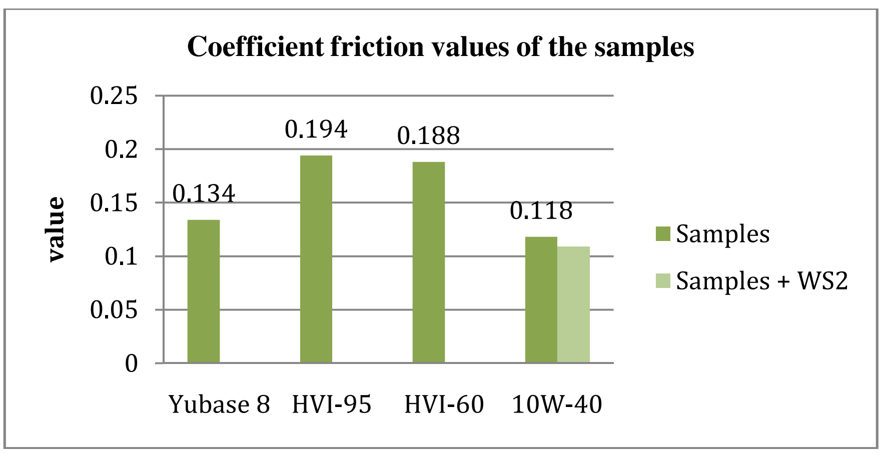


Fig.-6: The Coefficient Friction of Values of Base Oil Samples and its Combination with WS_2

Figure-6 gives a positive trend by reducing the friction coefficient of SAE 10W-40 lubricants after the addition of WS_2Nano additives. This can be seen by comparing W friction coefficient (lubricant without WS_2 Nano additives) and sample W-0.1 (lubricant with WS_2 Nano additives).

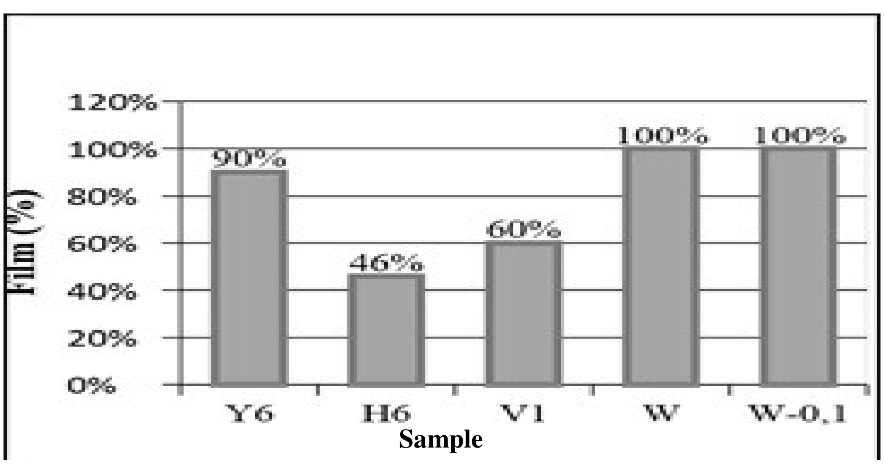


Fig.-7: Film Layer of HFRR Test

Profile test of lubrication film is prepared by merging profile parameter of all tested sample. It is shown in Fig.-7 below.

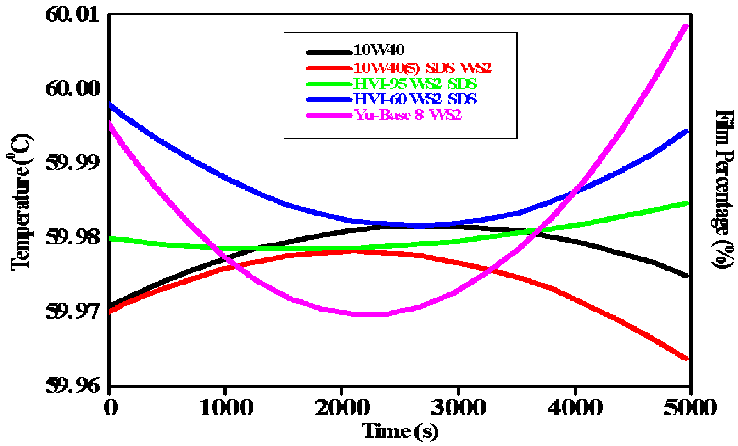


Fig.-8: Merged Profile Graph of HFRR Test on Film Lubrication

Figure-8 shows the lubrication film profile versus time. W and W-0.1 samples are seen to have stable lubrication even from the beginning of the run-in period. This indicates the lubricant is evenly distributed, covering the metal during alternating frequency in the study. In sample Y6, however, lubrication film started to get stable after 50 minutes of run in whereas sample H6 and V1 had relatively unstable lubrication from the beginning until the end of the test.

CONCLUSION

After Four-Ball and HFRR tests have been conducted, we can observe that the addition of WS_2 Nanoparticles is able to reduce friction force that takes place. This is shown in the decreasing of wear scar diameter in every sample as illustrated in Figure 2 which there is a decreasing trend in every addition of the WS_2 additives.Based on the study done, we know the optimum concentration for Tungsten Disulfide is 0.1% wt. and 0.2% wt. of Sodium Dodecyl Sulfate (SDS) surfactant for every sample. The test result of scar diameter for optimum composition for Yubase 8 based lubricant sample is 0.29 mm whereas for HVI-95 based is 0.33 mm whilst HVI-60 is 0.31mm. Thediameter of the samples decreased which accounted for 38.2%, 54.7% and 56.9% for every sample. Meanwhile, WS_2 additives added to multigrade SAE 10W-40 lubricant affects the protection characteristic to wear and tear and friction coefficient. Four-Ball scar diameter test for the sample with 0.1% wt. WS_2 added is 0.28 mm and 0.25 mm. While HFRR wears scar for multigrade SAE 10W-40 lubricant is 183.5 µm with 0.118 friction coefficient. By having sample added with 0.1% wt. of WS_2 the wear scar and friction coefficient become 108.5 and 0.109 respectively. The lubricating film on HFRR test for both samples before and after Nano additive is added is 100%.

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