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# Improvement of Lithium Grease Tribological Performance with Graphite as an Additive

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**Abstract**: By conducting experiments in accordance with the ASTM D 2596 standard as well as the ASTM 2266 standard, respectively, it was possible to determine how graphite powder's use as an intense pressure additive as well as an anti-wear additive affected the tribological characteristics of lithium grease. In order to determine the thresholds for ideal weld load as well as wear scar diameter, signal to noise ratio analysis also performed. According to a signal-to-noise ratio analysis, graphite powder of particle sizes of 0.5 micron and 5% by volume in lithium grease showed the greatest load carrying capacity compared to other combinations, and graphite powder of particle sizes of 5% by volume and 1.5 micron in lithium demonstrated the smallest wear scar diameter compared to other combinations. Weld load and worn scar diameter significant factors were found by analysis of variance. According to the analysis of variance, graphite volume percentage contributed more than graphite particle size to weld load and graphite volume percentage contributed more than graphite particle size to wear scar diameter. When compared to ordinary lithium grease, it was found that graphite with lithium grease at ideal concentrations showed greater weld and smaller wear scar diameter.

**Keywords**: Anti wear additive, Analysis of Variance, ASTM D 2266, ASTM D 2596, Four ball Tester, Extreme Pressure Additive, Signal to Noise ratio.

## 1. Introduction

Grease is a mixture of lubricating oils as well as soaps or thickeners that is semi-solid in consistency. Commonly used thickeners include lithium, calcium, sodium, and aluminium soaps. Although greases are frequently used as lubricants, they are unable to meet the lubrication requirements for all applications without additions. Graphite can be used as a grease addition for intense pressure and anti-wear for strongly loaded applications. When operating a bearing, the grease should be thick enough to support the load and shouldn't thin out [3]. Lithium soap grease is water and oxidation resistant. At high temperatures, the lithium soap grease has good shear stability.

The findings described by Bartz [1] that an ideal concentration of molybdenum di sulphide with graphite exists in liquid or paste lubricants represents one of the most significant attempts to identify graphite as an addition in grease. This level of solid lubricant concentration is necessary to sustain wear resistance. The creation of an entire coating shielding the surfaces will determine how well solid lubricants work to lubricate surfaces. According to Chu et al. [2], graphite formed composition films also with oil-soluble additives and was observed to be present upon that rubbing surfaces steadily. Additionally, Antony et al. [3] reported research on the anti-wear and severe pressure performance of greases containing mixtures including graphite and molybdenum di sulphide. The anti-wear and severe pressure properties of organo clay and lithium base greases are enhanced both by molybdenum di sulphide and graphite separately. In terms of severe pressure and anti-wear properties, it has been discovered that molybdenum di sulphide and graphite can work together synergistically. The two component ratios and grease type affect the synergism, though. According to Huang et al. [4], the inclusion of graphite nanosheets can increase the paraffin oil's load carrying and wear resistance ability while lowering its friction coefficient. The lubricating oil contains the ideal amount of graphite nanosheets, providing the highest maximum nonseized antiwear and load capabilities.

As lubricant additives, poly tetrafluoroethylene (PTFE), molybdenum disulfide, and titanium oxide had their tribological properties studied by Reick [5, Gansheimer et al. [6, and Hu et al. [7]. Tribological characteristics are influenced by lubricant additives [8–12]. Lubricant additives with nanoparticle sizes improve tribological performances as well [13, 14].

It was discovered from the literature that the majority of research on the effects of graphite as an addition in greases was based on the graphite's optimum concentration for greases and liquid lubricants. The purpose of this research was to determine the ideal graphite content and particle size to add to lithium grease for the best load carrying ability and anti-wear performance. In varying volume percentages of 5%, 10%, and 15%, lithium soap grease was combined with graphite particles of 0.5 micron, 1 micron, and 1.5 micron diameters. Weld load and wear scar diameter were measured using a four-ball tester in accordance with the ASTM D 2266 and ASTM D 2596 standards, respectively.

## 2. Experimentation

### 2.1 Lithium Grease

The appearance of lithium soap grease is either smooth or grainy. Lithium soap grease offers high temperature properties similar to sodium soap grease and water resistant properties similar to calcium soap grease.

### 2.2 Graphite Selection as an additive

In greases, additives can strengthen the already-present favourable qualities or reduce the already-present negative features while introducing new ones. Because inorganic compounds have high tribological properties, inorganic compounds are preferred when choosing an additive. The other most crucial factor, in addition to inorganic compounds, is that solid lubricant has better tribological qualities under adverse circumstances. Graphite was chosen as an intense pressure and anti-wear addition based on the research's goals.

### 2.3 Experimental Design

The Taguchi approach served as the foundation for the design of the tests. This allowed for the achievement of results with fewer experiments. This study aimed to determine the impact of graphite content and particle size on the lithium grease's performance under anti-wear conditions and extreme pressure (EP). As a result, lithium grease has a combination of 5%, 10%, and 15% of graphite by volume and comes in size of particles of 0.5, 1, and 1.5 micron. Table 1 lists the factors and their corresponding levels.

**Table 1. Factors and their levels**

| Factor | 1st Level | 2nd Level | 3rd Level |
|---|---|---|---|
| Size of Particle (micron) | 0.50 | 1.00 | 1.50 |
| Percentage proportion (by volume) | 5.00 % | 10.00 % | 15.0 % |

## 2.4 Four Ball- Tester Machine

Tests were conducted using four ball tester machine (TR-30L-IAS0) made by DUCOM. The schematic loading configuration used in the testing is shown in Figure 1. The weld loads as well as wear scar diameter of pure lithium grease and pure lithium grease including graphite powder as an addition were determined using the ASTM D 2596 as well as ASTM D 2266 standard procedures, respectively. Three balls are securely held in the ball pot assembly at the bottom of the four ball tester machine, and one ball is securely held in the spindle collet at the top. Three balls and a sample of grease were placed in the ball pot assembly. The spindle's rotational speed was adjusted.

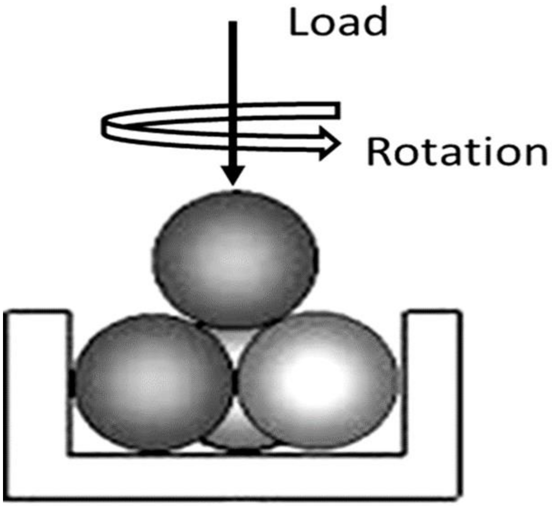


**Figure 1. Schematic Loading Arrangement in Four Ball Tribometer**

## 2.5 Test Preparation

Samples of grease were created in accordance with the experiment's plan. In accordance with the volume capacity of the ball pot, 18 milliliter of lithium grease and three different particle sizes and percentage proportions of graphite powder were added. Acetone is used to clean the ball pot, collets, and four balls. According to Figure 2, three balls were kept in a ball pot and secured by a locking ring. The ball pot was entirely filled with the grease sample. When placing the grease sample in the ball pot, attention was paid to remove any air pockets. Ball pot assembly was cleaned of extra grease. The top ball was put on the spindle after being mounted in the collet. The friction disc was set atop the ball pot.

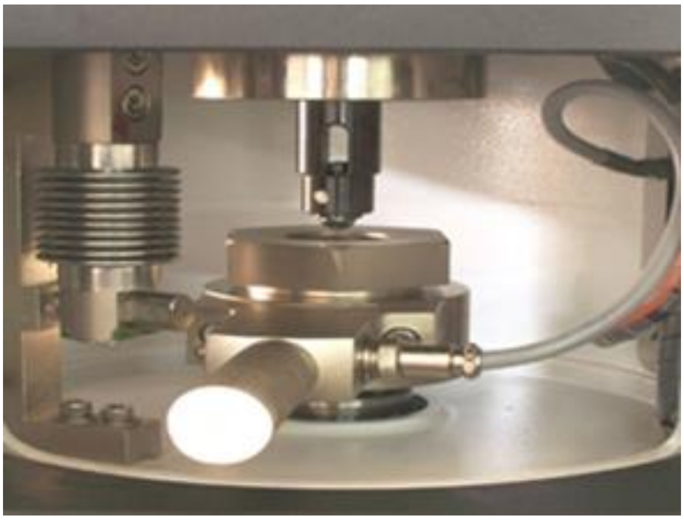


**Figure 2. Ball Pot Assembly**

## 2.6 Tribological Test

### 2.6.1 As per ASTM D 2596Testing -Ext. Pressure Tests [15]

Testing was carried out in accordance with ASTM D 2596 standard practises to assess weld load. For the experiments, 12.7 mm diameter carbon chrome balls were employed. A regular speed of 1770 rpm and a temperature of 27 8 oC were maintained. A load lever was used to apply the required load. Three balls in the ball pot were in touch with the top ball as it was being rotated by the spindle. The motor was driven for the prescribed 10 seconds. Motor noise or a fast drop in pressure might also indicate a weld spot. If the balls are not fused, the load is regarded as "passed." In such a scenario, the following load in accordance with specifications was applied, and the test method was repeated until weld load was attained. Table 2 contains the weld loads for the nine experiments.

**Table 2. Result table and L9 orthogonal array**

| **Test number** | **Factor** | | **ASTM D2596 Extreme pressure Tests** | | **ASTM D 2266 Anti-wear Tests** | |
|---|---|---|---|---|---|---|
| | **Size of Particle (micron)** | **Proportion (by volume) (%)** | **Weld load (kg-f)$^*$** | **S/N ratio** | **Diameter Avg. wear scar (microns)** | **S/N ratio** |
| - | Plain Lithium grease Sample | | 400 | -- | 753 | -- |
| 1 | 0.5 | 5 | 620 | 55.8478 | 796 | -58.0183 |
| 2 | 0.5 | 10 | 500 | 53.9794 | 938 | -59.4441 |
| 3 | 0.5 | 15 | 400 | 52.0412 | 1167 | -61.3414 |
| 4 | 1 | 5 | 500 | 53.9794 | 698 | -56.8771 |
| 5 | 1 | 10 | 315 | 49.9662 | 812 | -58.1911 |
| 6 | 1 | 15 | 250 | 47.9588 | 1019 | -60.1635 |
| 7 | 1.5 | 5 | 315 | 49.9662 | 651 | -56.2716 |
| 8 | 1.5 | 10 | 250 | 47.9588 | 749 | -57.4896 |
| 9 | 1.5 | 15 | 200 | 46.0206 | 956 | -59.6092 |

### 2.6.2 As per ASTM D 2266 Testing (Anti-wear tests) [16]

Testing was carried out in accordance with ASTM D 2266 standard standards, and carbon chrome balls with a diameter of 12.7 mm each were employed in the tests. Both the temperature and the speed were kept within the prescribed ranges of 75 2 oC and 1200 60 rpm. The load lever applied the required load of 392 N. Three balls in the ball pot were in touch with the top ball as it was rotated by the spindle. The motor was ran for the prescribed 60 minutes. Following the test, the image acquisition system assessed the wear scar sizes for three balls in the ball pot in microns. The widths of typical wear scars are listed in Table 2. A L9 orthogonal array with 9 experiments and three tiers of two factors is shown in Table 2. With nine experiments, the orthogonal array has 8 degrees of freedom.

## 3. Results

### 3.1 S/N ratio (Signal to noise) analysis

When noise elements are present, the signal to noise ratio—a measure of fluctuation within the trial—is taken into account as an experiment's response [17].

According to ASTM D 2596 standards, weld load was taken into consideration as the reaction. The "larger-is-better" criterion was taken into consideration because the grease can carry more load when the weld load is greater. Equation calculated the S/N ratio (1)

$$\frac{S}{N} = -10\log_{10}\frac{1}{n}\sum_{i=1}^{n}\frac{1}{{y_i}^2} \qquad (1)$$

Where n represents the number of experiments and yi is the number of response values in the tests. Table 2 contains the S/N ratio data. According to Figure 3, adding graphite at levels 1 of particle size (0.5 microns) and percentage proportion (5%) to lithium grease will produce the best weld load.

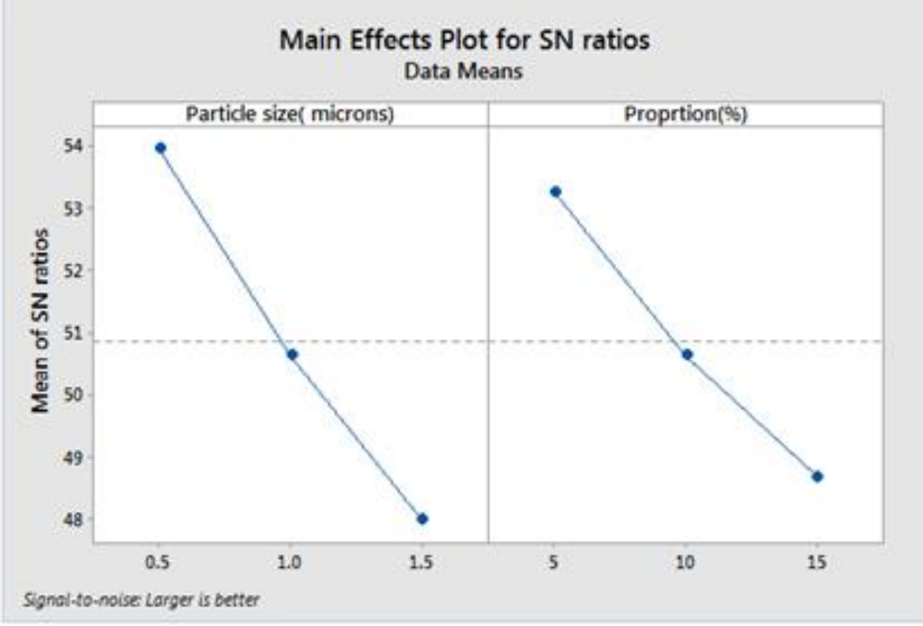


**Figure 3. Factors Effect on S/N ratio - weld load**

According to ASTM D 2266 standards, the wear scar diameter was taken into consideration. Because grease has wear-preventive properties, the smaller the wear scar diameter, the better; hence, the "smaller-the-better" criterion was taken into consideration. Equation calculated the S/N ratio (2).

$$\frac{S}{N} = -10\log_{10}\frac{1}{n}\sum_{i=1}^{n} {y_i}^2 \qquad (2)$$

Where yi is the total number of test responses, and n is the total number of experiments.
Table 2 contains the values for the S/N ratio. Figure 4 suggests that adding graphite at levels 3 of particle size (1.5 microns) and 1 of percentage (5%) will produce lithium grease with the best wear scar diameter.

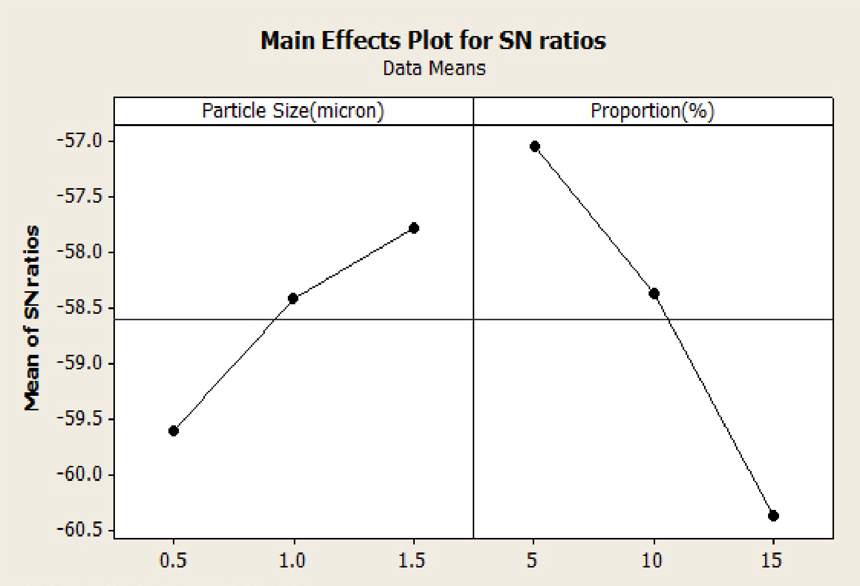


**Fig 4. Factors Effect on S/N ratio - average wear scar diameters**

To determine the impact of the each levels of particle size and % share on weld load and typical wear scar diameters, the average S/N ratios shown in Table 3 in tests 1 through 9 are examined. The weld load as well as wear scar diameter are highly influenced by factors with a substantial difference in the average S/N ratio. According to Table 3, the percentage proportion has a greater impact on wear scar diameter than does the particle size on weld load.

**Table 3. Average S/N ratio Analysis for weld loads**

| Levels | Extreme pressure Tests ASTM D2596 | | Anti-wear Tests ASTM D 2266 | |
|---|---|---|---|---|
| | **Size of Particle (micron)** | **Volume Proportion (%)** | **Size of Particle (micron)** | **Volume Proportion (%)** |
| 1 | 53.95610 | 53.26440 | -59.600 | -57.060 |
| 2 | 50.63480 | 50.63480 | -58.410 | -58.370 |
| 3 | 47.98180 | 48.67350 | -57.790 | -60.370 |
| Δ = Max-Min | 5.9743 | 4.5909 | 1.81 | 3.32 |
| **Rank** | **1** | **2** | **2** | **1** |

### 3.2 ANOVA (Analysis of Variance)

The major component that influences welds load and wear scar diameter was tested using the analysis of variance as a statistical method. ANOVA was used to analyse how much each factor contributed to the welds load and wear scar diameter. Variance ratio, often known as the F statistic, is the proportion of variance attributable to a factor's effects to variance attributable to error terms. F value establishes the relevance of a factor. The probability value, or p value, establishes the amount of relevance of each element. A greater impact on the experiment's outcome is indicated by a factor with a lower p value. When a factor has a large S/N ratio, it typically has a lower p value, which means it has a greater influence on the experiment's results. Table 4 lists the Minitab 17 software's ANOVA findings for tests under extremely high pressure.

**Table 4. ANOVA -Lithium grease and Graphite as an additive weld loads**

| Parameter | DOF | SS | MS | F-Value | P-Value | P % |
|---|---|---|---|---|---|---|
| Size of Particle (microns) | 2 | 96339 | 48169 | 31.10 | 0.004 | 59.87 |
| Percentage Proportion | 2 | 58372 | 29186 | 18.85 | 0.009 | 36.27 |
| Residual Error | 4 | 6194.0 | 1549 | | | 3.840 |
| Total | 8 | 160906. | | | | 100 |
| S = 39.35240 R-Sq = 96.150% R-Sq(adj) = 92.300% | | | | | | |

The lower p value in Table 4 for graphite's particle size implies that it has a greater impact on weld load. Figure 5 demonstrates that graphite's additional particle size contributed significantly more to the weld load—by 59.87%—than graphite's percentage contribution, which was only 36.27%.

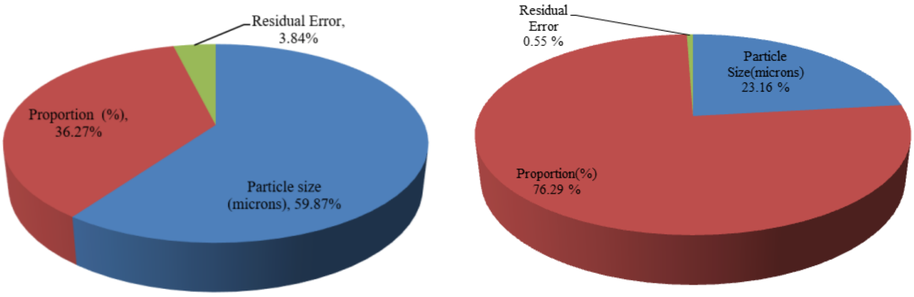


**Figure 5. Factor Contribution on weld loads**

Table 5 contains the anti-wear test ANOVA results. Since graphite has a lower p value than other materials, it is likely to have a greater impact upon wear scar diameter. Figure 6 illustrates how graphite's additive percentage fraction contributed more significantly to wear scar diameter (76.29%) than graphite's 23.16% particle size.

**Table 4. ANOVA- Lithium grease and Graphite as an additive for wear scar diameters**

| Parameter | DOF | SS | MS | F-Value | P-Value | P % |
|---|---|---|---|---|---|---|
| Size of Particle (microns) | 2 | 51704 | 25852 | 84.18 | 0.001 | 23.16 |
| % Proportion | 2 | 170308 | 85154 | 277.270 | 0.000 | 76.290 |
| Resi. Error | 4 | 1228 | 307 | | | 0.550 |
| Total | 8 | 223241 | | | | 100 |
| S = 17.52460 R-Sq = 99.450% R-Sq(adj) = 98.900% | | | | | | |

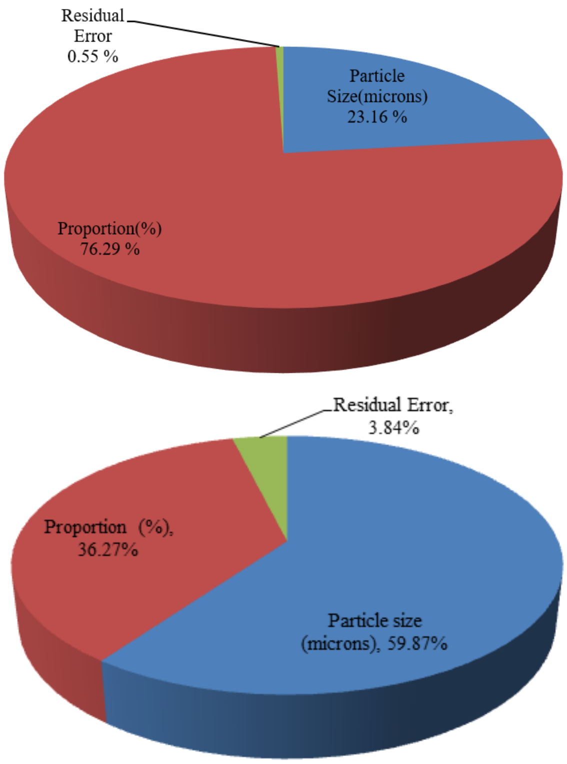


**Figure 6. Factor Contribution on wear scar diameters**

### 3.3 Factor Effect Confidence Interval

The population parameters are only given as estimates in the ANOVA table. The size of the study's sample will determine this estimate. The best experiment outcome must therefore be predicted with a degree of confidence. The confidence interval (CI), for such a given level of confidence, is the range that a statistical parameter falls within. Calculating the CI yields [18]

$$CI = \pm\sqrt{\frac{F_\alpha(f_1,f_2)*v_e}{n_e}} \qquad (3)$$

Here,

$F_\alpha(f_1, f_2)$ is Variance ratio for degree of freedom (DOF) $f_1$ and $f_2$ at the level of significance.

$f_1$ is DOF of mean (always 1)

$f_2$ is DOF of error term= 4

For CI 90%, $F_\alpha(1.4)\ is$ 4.54

Ve is Variance of error term

For ASTM D 2596 test from Table 4, Ve = 1549

For ASTM D 2266 test from Table 5, Ve = 307

$n_e = Number\ of\ equivalent\ replications$

$$n_e = \frac{Number\ of\ Trials\ (n = 9)}{DoF\ of\ mean\ (always\ 1) +\ DoF\ of\ the\ Factors\ used\ in\ Estimate} \tag{4}$$

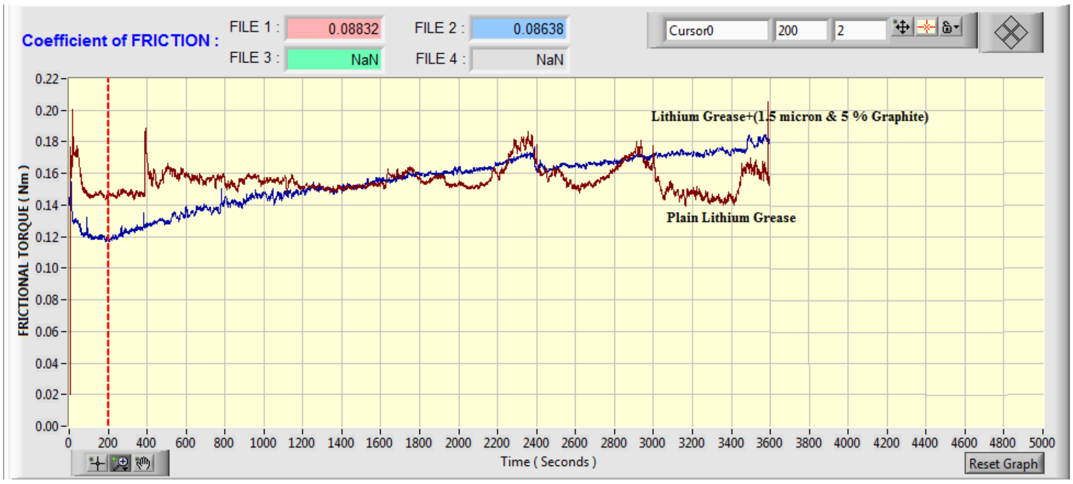
$$n_e = 1.8$$

The ideal ASTM D 2596 test factors (particle size 0.5 micron, 5% graphite as an additive) were confirmed after three tests. The 90% CI includes the average load value (620 kg-f). At a 90% level of confidence, the computed CI (m) = 62.50 kg-f. In light of this, the estimated weld load at the ideal situation, at a 90% confidence level, is 620 62.50 kg-f.

For the best ASTM D 2266 test conditions (1.5 micron particle size, 5% addition graphite), 3 confirmation tests were carried out. The 90% confidence interval includes the mean value of worn scar diameter (640.66 microns). At a 90% level of confidence, the computed CI (m) = 27.82 microns. As a result, the estimated wear scar diameter in the ideal condition is, at a 90% confidence level, 640.66 27.82 microns.

## 4. Comparison of Weld Load, Frictional Behavior and Wear Scar Diameter of Lithium Grease and Plain Lithium Grease with Graphite Optimal Levels

Table 2 shows the average wear scar diameter and weld load of plain lithium grease. At confirmation test, the weld load at factors optimal levels (0.5 micron and 5 % of graphite) is 620 kg-f. There is about 35.48 % of increase in weld load as compared to plain lithium grease. Lithium grease has the required yield strength for effective lubrication due to the graphite's smaller particle size in the grease. It produces a thin layer on the surface and can attach to metal surfaces sufficiently because of its affinity for metal. In comparison to plain lithium grease, lithium grease containing graphite has a higher load-carrying capacity because its high yield strength is maintained in the direction opposite to the direction of shear force.



Coefficient of FRICTION :
FILE 1 : 0.08832
FILE 2 : 0.08638
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Figure 7. Comparison of frictional torque of plain Lithium grease and 1.5 micron Lithium Grease and 5 % of graphite

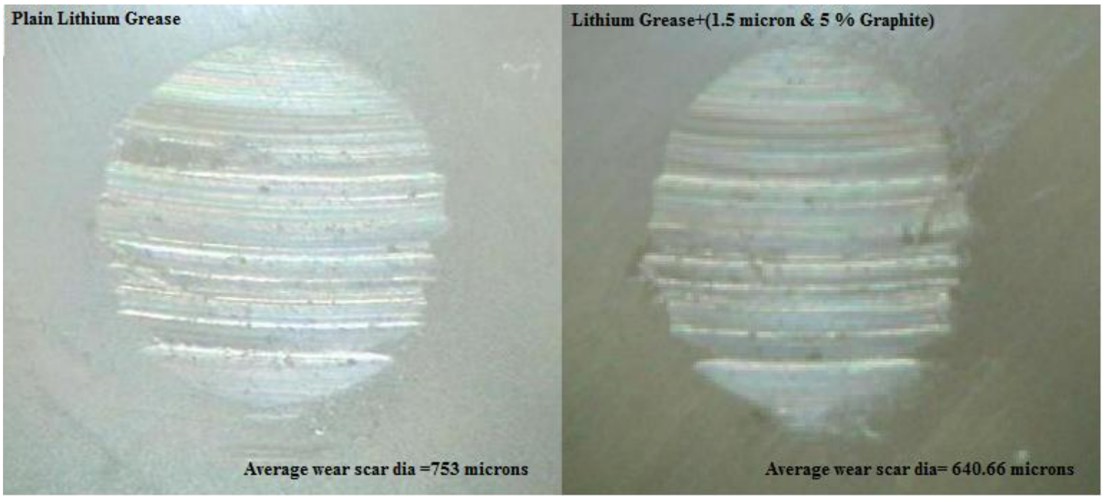

**Figure 8. Comparison of plain Lithium grease and Lithium Grease with 1.5 micron and 5 % of graphite wear scar diameter**

At the confirmation test, its average wear scar diameter is 640.66 microns at the ideal factor concentrations (1.5 micron and 5% graphite). In comparison to plain lithium grease, the average wear scar diameter has decreased by roughly 14.91%. The structure of the graphite is lamellar. The applied shear force runs parallel to the sliding direction. Graphite planes can be easily sheared in the force's direction due to the weak link between them. As a result, the planes slide and there is less friction. Therefore, average wear scar diameter is reduced for lithium grease with larger particle size graphite. From Table 2, the average wear scar diameter seems to increase at higher concentrations due to increase in internal friction between sliding of layers of graphite. Figure 7 compares the frictional torque and coefficient friction of lithium grease with and without graphite particles of 1.5 microns in concentration. Compared to lithium grease with 1.5 micron and 5% graphite, the frictional torque for lithium grease is larger at first. In comparison to lithium grease with 1.5 micron and 5% graphite, plain lithium grease has a greater coefficient of friction. The average wear scar sizes of lithium grease with 1.5 microns and 5% graphite and lithium grease in plan are shown in Figure 8.

## 5. Conclusion

The experimental investigation looked at how the amount and percentage of graphite added to lithium grease as an additive affected the grease's anti-wear and load bearing capacities. Conclusively, after summarising the key findings,

1. Based on the L9 orthogonal array developed by the Taguchi approach, nine tests were carried out to determine the weld load and wear scar diameters of the lithium grease with graphite as an anti-wear additive and extreme pressure, complying to ASTM D2596 and ASTM D 2266 requirements, correspondingly.
2. A higher S/N ratio for the weld load is preferred since lithium grease using graphite as an addition has a higher load carrying capacity. Test 1 (0.5 micron and 5% by volume of graphite with lithium grease) is the ideal combination, according to an examination of S/N ratios, with a higher S/N ratio.
3. The phrase "smaller is better" is applied to the S/N ratio for wear scar diameter because smaller wear scar diameters are preferable for demonstrating superior wear preventative characteristics of lithium grease using graphite as an addition. Test 7 (1.5 micron and 5% by volume of graphite with lithium grease) is the ideal combination, according to S/N ratio analysis, because it has the smallest S/N ratio.

4. S/N ratio analysis also demonstrated that weld load is decreased by increasing the percentage of graphite with lithium grease as well as by increasing the graphite's particle size. Similar to this, increasing the percentage of graphite within lithium grease reduces wear scar diameter while increasing the graphite's particle size increases wear scar diameter.
5. Mean S/N ratios for weld loads with wear scar widths at levels 1, 2, and 3 were calculated using averaging S/N ratios for tests 1 through 9 in order to determine the influence of each level of variables particle size as well as percentage proportion. According to this data, percentage proportion and particle size have greater effects on wear scar widths and weld load, respectively. Additionally, it was discovered that level 1 of the factors—particle size (0.5 microns) and percentage proportion (5%)—will produce the best weld load. The ideal wear scar diameter will be determined by level 1 of factor percentage proportion (5%) and level 3 of factor particle size (1.5 microns), respectively.
6. Graphite additive particle size contributed significantly more (59.87%) to weld load than percentage proportion by volume (36.27%), while percentage proportion by volume significantly more (76.29%) than graphite particle size (23.16%) to wear scar diameter, according to the findings of an ANOVA.
7. Testing were undertaken to confirm the appropriate factors (0.5 micron particle size, 5 percentage proportion additive) for ASTM D 2596 tests. Within a 90% confidence interval, the average load value (620 kg-f) is found. The expected weld load is 620 62.50 at 90% certainty (kg-f).
8. Confirmation tests for the ideal components (1.5 micron particle size, 5 percentage proportion additive) were carried out for the ASTM D 2266 tests. The forecast weld load at 90 % confidence interval is 640.66 ± 27.82 microns.
9. In the confirmation test, the weld load is 620 kg-f at the optimal factor levels (0.5 micron and 5% graphite). When compared to normal lithium grease, the weld load increases by around 35.48%. At the confirmation test, the average wear scar diameter is 640.66 microns at the ideal factor concentrations (1.5 micron and 5% graphite). In comparison to plain lithium grease, the average wear scar diameter has decreased by roughly 14.91%.
10. Compared to lithium grease using 1.5 micron and 5% graphite, the frictional torque for lithium grease is larger at first. In comparison to lithium grease using 1.5 micron and 5% graphite, plain lithium grease has a greater coefficient of friction.

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