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A Taguchi Approach on Influence of Graphite as an Anti-Wear Additive on the Performance of Lithium Grease

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Abstract

An Influence of graphite powder as an extreme anti-wear additive on the tribological performance of lithium grease was identified by conducting tests as per ASTM 2266 standard. Signal to noise ratio analysis was done to identify the levels for optimum wear scar diameter. Analysis of variance was done to identify significant factor which affects wear scar diameter. For optimum levels, wear scar diameter was predicted.

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1. Introduction

Greases are semi-solid substances composed of lubricating oils and soaps or thickeners. Soaps of lithium, calcium, sodium, aluminum are commonly used thickeners. Greases are popularly used as lubricant; however without additives greases cannot fulfill particular application lubrication requirements. For heavily loaded applications graphite can be effectively used as an extreme pressure and anti-wear additive in greases. The grease should have consistency to carry load during bearing operation and should not thin during entire operation cycle [3]. The lithium soap grease is resistant to water and oxidation. The lithium soap grease shows good shear stability at high temperature.

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One of the most important attempt in identifying graphite as an additive in grease was the work reported by Bartz [1] that optimal concentration exist for molybdenum di sulphide and graphite in liquid or paste lubricants. Less than this concentration of solid lubricant is insufficient to maintain protection against wear. The effectiveness of lubrication with solid lubricants will depend on the formation of a complete film protecting the surfaces. Chu et al. [2] found that graphite existed on the rubbing surfaces stably and formed composition film with the oil-soluble additives. Further, Antony et al. [3] reported work on anti-wear/extreme pressure performance of graphite and molybdenum di sulphide combinations in lubricating greases. Both molybdenum di sulphide and graphite individually improve the anti-wear and extreme pressure characteristics of lithium and organo clay base greases. Combinations of molybdenum di sulphide and graphite had been found to exhibit synergistic effect in extreme pressure and anti-wear characteristics. However, the synergism depends on ratio of two components and type of grease. Huang et al. [4] reported that the wear resistance and load carrying capacity of paraffin oil can be improved and its friction coefficient can be decreased by the addition of the graphite nanosheets. There is an optimal content of graphite nanosheets in the lubricating oil, which gives the highest maximum nonseized load and antiwear ability.

The tribological properties of poly tetra fluoro ethylene (PTFE) by Reick [5], molybdenum disulfide by Gansheimer et al. [6], titanium oxide by Hu et al. [7] as lubricant additives had been investigated. Additives in lubricants influence tribological properties [8-12]. Nano particles size lubricant additives also enhance tribological properties [13, 14].

From literature, it was found that most of study of influence of graphite as an additive in greases was based on optimal concentration of graphite in liquid lubricants and greases. The aim of this work was to identify effect of concentration and effect of particle size of graphite as an anti- wear additive in lithium grease for the optimum wear scar diameter. The particle sizes of 0.5 micron, 1 micron and 1.5 micron of graphite was mixed in lithium soap grease in different volume proportions of 5%, 10% and 15%. A four ball tester was used to measure wear scar diameter by using ASTM D 2266 standard.

2. Experimental Study

2.1 Lithium Grease

Lithium soap grease is smooth or granular in appearance. Lithium soap grease provides water resistant property like calcium soap grease and high temperature property like sodium soap grease.

2.2 Selection of Graphite as an additive

For greases, additives enhance the existing desirable properties or suppress the existing undesirable properties and impart new properties. During selection of additive it is considered that the additive should be inorganic compound because inorganic compound shows excellent tribological properties. The other most important consideration is that solid lubricant shows better tribological properties at extreme conditions. From the objectives of research graphite was selected as an anti-wear additive.

2.3 Design of Experiments

Design of experiments was done on the basis of Taguchi technique. By this way, it was possible to achieve results with less number of experiments. The purpose of this research was to identify effect of particle size and concentration of graphite by volume in lithium grease on extreme pressure (EP) and anti-wear performance of the lithium grease. Therefore, particle sizes of 0.5 micron, 1 micron and 1.5 micron of graphite are used along with combination of 5%, 10% and 15% proportion of graphite by volume in lithium grease. Factors and their respective levels are listed in Table 1.

Table 1. Factors and their levels

Factors	Level 1	Level 2	Level 3
Particle size (micron)	0.5	1	1.5
Percentage proportion by volume	5 %	10 %	15 %

Table 2 shows L9 orthogonal array consisting of nine experiments and three levels of two factors. The orthogonal array with nine experiments has 8 degrees of freedom.

2.4 Four Ball Tester Machine

The four ball tester machine TR-30L-IAS DUCOM as shown in Fig.1. make was used for tests. ASTM D 2266 standard procedure was followed to find wear scar diameter respectively of lithium grease with graphite powder as an additive. Four ball tester machine consist of three balls held firmly in ball pot assembly at bottom and one ball at top held in spindle collet. Grease sample was put in ball pot assembly along with three balls. The spindle was made to rotate at desired speed.



Fig.1.Four Ball Tester

2.5 Preparation of Test

Based on design of experiments grease samples were prepared. Approximately 18 ml of lithium grease was added with graphite powder with three particle sizes and three percentage proportions as per volume capacity of ball pot. Four balls, ball pot, collets are cleaned using acetone. Three balls were held in ball pot and locked by a locking ring as shown in Fig. 2. The grease sample was completely filled in ball pot. Care was taken to avoid air pockets while filling grease sample in ball pot. The additional grease was removed from ball pot assembly. The top ball was inserted in collet and mounted on spindle. The ball pot was placed on the antifriction disc.



Fig.2 Ball Pot Assembly

2.6 Tribological Testing As Per ASTM D 2266

To determine wear preventive characteristics, testing was done as per ASTM D 2266 standard procedures, carbon chrome balls of 12.7 mm diameter each were used in tests. The temperature of 75 ± 2 °C and speed of 1200 ± 60 rpm were maintained as per standards. The load 392 N as per standard was applied by load lever. The top ball was rotated by the spindle in contact with three balls in ball pot. The motor was run for 60 minutes duration as per standards. After the test, wear scar diameters for three balls in ball pot were measured using image acquisition system in microns. Average wear scar diameters are listed in Table 2.

Table 2. L_9 orthogonal array and result table

Test No.	Particle size (micron)	Proportion by volume (%)	Avg. wear scar diameter (microns)	S/N ratios
1	0.5	5	796	-58.0183
2	0.5	10	938	-59.4441
3	0.5	15	1167	-61.3414
4	1	5	698	-56.8771
5	1	10	812	-58.1911
6	1	15	1019	-60.1635
7	1.5	5	651	-56.2716
8	1.5	10	749	-57.4896
9	1.5	15	956	-59.6092

3. Results and Discussion

3.1 Signal to Noise (S/N) Ratio Analysis

Smaller the wear scar diameter better is wear preventive characteristics of grease, therefore ‘the smaller-the-better’ criterion was considered.

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

Where, y_i is number of response value (wear scar diameters) in the tests and n is the number of experiments. S/N ratio values are listed in Table 2. Fig. 3. shows that level 3 of particle size (1.5 micron) and level 1 of percentage proportion (5 %) of graphite as an additive in lithium grease will give optimum wear scar diameter.

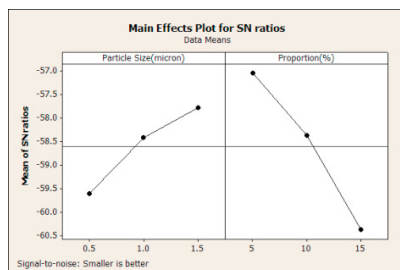


Fig.3. Effect of factors on S/N ratio for average wear scar diameters

Average S/N ratios listed in Table 3 shows influence of each level of particle size and percentage proportion on weld load and average wear scar diameters. Factor with large difference (Δ) of average S/N ratio means high influence wear scar diameter. Table 3 suggests that percentage proportion has larger influence on wear scar diameter.

Table 3. Analysis of average S/N ratios

Levels	Particle size (micron)	Proportion by volume (%)
1	-59.60	-57.06
2	-58.41	-58.37
3	-57.79	-60.37
$\Delta = \text{Max} - \text{Min}$	1.81	3.32
Rank	2	1

3.2 Analysis of Variance (ANOVA)

The analysis of variance was used as statistical tool for testing the significant factor which affects weld load and wear scar diameter. The ratio of variance due to effects of a factor and variance due to error term is called variance ratio or F statistic. The significance of factors is determined by F value. The level of significance of each factor is determined by p value or probability value. Factor with lower p value indicate that the factor has larger impact on the outcome of experiment. Generally, factors with large S/N ratio have low p value, which indicates that the factor has larger impact on the outcome of experiment. The ANOVA results tabulated for anti-wear are given in Table 4.

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

Factors	DOF	SS	MS	F-Value	P-Value	P %
Particle size (microns)	2	51704	25852	84.18	0.001	23.16
Percentage Proportion	2	170308	85154	277.27	0.000	76.29
Residual Error	4	1228	307			0.55
Total	8	223241				100
S = 17.5246 R-Sq = 99.45% R-Sq(adj) = 98.90%						

Fig.4. shows, for graphite as an additive percentage proportion gave more significant contribution of 76.29 % than particle size of graphite 23.16 % on wear scar diameter.

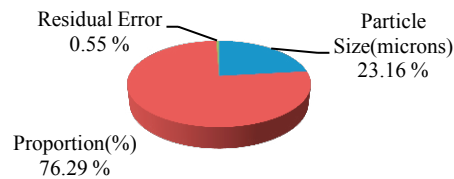


Fig.4. Percentage Contribution of factors for average wear scar diameters

3.3 Confidence Interval of Factor Effect

It is necessary to predict optimum outcome of experiment with some confidence interval. Confidence interval (CI) [18] is the range within which a statistical parameter falls, for a given level of confidence. The CI is calculated as

$$CI = \pm \sqrt{\frac{F_{\alpha}(f_1, f_2) \times V_e}{n_e}} \quad (2)$$

Where,

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

f_1 = DOF of mean (which is always 1)

f_2 = DOF of error term = 4

For 90% CI, = 4.54

V_e = Variance of error term

For anti-wear tests from Table 4, $V_e = 307$

n_e = number of equivalent replications

$$= \frac{\text{Number of Trials (n = 9)}}{\text{DOF of mean (always 1) + DOF of the factors used in estimate (= 4)}}$$

Three confirmation tests were conducted for optimal levels of factors (1.5 micron particle size, 5 % graphite as an additive) for anti-wear test. The average value of wear scar diameter (640.66 microns) falls within 90% CI. The calculated CI is ± 27.82 microns at 90 % confidence level. Therefore, the predicted wear scar diameter at the optimum condition is 640.66 ± 27.82 microns at 90% confidence level.

4. Conclusion

Summarizing main features of results as follows conclusively,

1. Smaller wear scar diameter is desirable for exhibiting better wear preventive characteristics of lithium grease with graphite as an additive; therefore ‘smaller is better’ is applied for S/N ratio for wear scar diameter. S/N ratio analysis revealed that test 7 (1.5 micron and 5 % proportion by volume of graphite in lithium grease) is an optimum combination.
2. Average S/N ratio analysis suggested that level 3 of factor particle size (1.5 micron) and level 1 of factor percentage proportion (5 %) respectively will give optimum wear scar diameter.
3. ANOVA results revealed that for graphite as an anti- wear additive percentage volume proportion gave more significant contribution of 76.29 % than percentage volume proportion of graphite 23.16 % on wear scar diameter.
4. For optimal factors (1.5 micron particle size, 5 percentage volume proportion additive), the predicted wear scar diameter at 90 % confidence interval is 640.66 ± 27.82 microns.

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References

- [1] W.J. Bartz, Solid lubricant additive-effect of concentration and other additives on antiwear performance, *Wear* 17 (1971) 421–432.
- [2] Shufeng Chu, Zhishan Jin, Qunji Xue, Study of the interaction between natural flake graphite and oil soluble additives, *Tribology International* 17 (4) (1997) 340–347.
- [3] J.P. Antony, B.D. Mittal, K.P. Naithani, A.K. Misra, A.K. Bhatnagar, Antiwear/extreme pressure performance of graphite and molybdenum disulphide combinations in lubricating greases, *Wear* 174 (1994) 33–37.
- [4] H.D. Huang, J.P. Tu, L.P. Gan, C.Z. Li, An investigation on tribological properties of graphite nanosheets as oil additive, *Wear* 261 (2006) 140–144.
- [5] F.G. Reick, Energy saving lubricants containing colloidal PTFE, *Lubrication Eng.* 38 (1982) 635–646.
- [6] J. Gansheimer, R. Holinsk, Molybdenum disulfide in oils and greases under boundary conditions, *ASME Journal of Lubrication Technology* 95(1973) 242–248.
- [7] Z.S. Hu, J.X. Dong, Study on antiwear and reducing friction additive of nanometer titanium oxide, *Wear* 216 (1998) 92–96,
- [8] I.M. Petrushina, E. Christensen, R.S. Bergqvist, P.B. Moller, N.J. Bjerrum, J. Hoj, G. Kann, I. Chorkendorff, On the chemical nature of boundary lubrication of stainless steel by chlorine and sulfur containing EP additives, *Wear* 246 (2000) 98–105.
- [9] P. Waara, J. Hannu, T. Norrby, A. Byheden, Additive influence on wear and friction performance of environmentally adapted lubricants, *Tribology International* 34 (2001) 547–556.
- [10] A. Neyman, Comparative investigation of lubricant properties in friction and wear tests, *J. of KONES Internal Combustion Engines* (2002) 204–210.
- [11] R.B. Rastogi, M. Yadav, Suspension of molybdenum sulphur complexes in paraffin oil as extreme pressure lubricants, *Tribology International* 36 (2003) 511–516.
- [12] M.H. Choa, J. Jeong, S.J. Kima, H. Jang, Tribological properties of solid lubricants (Graphite, Sb₂S₃, MoS₂) for automotive brake friction materials, *Wear* 260 (2006) 855–860.
- [13] M. Alberts, K. Kalaitzidou, S. Melkote, An investigation of graphite nano platelets as lubricant in grinding, *International journal of Machine Tools & Manufacturing* 49 (2009) 966–97.
- [14] Y.Y. Wu, W.C. Tsui, T.C. Liu, Experimental analysis of tribological properties of lubricating oils with nanoparticle additives, *Wear* 262(2007) 819–825.
- [15] ASTM D 2596. Standard test method for measurement of extreme-pressure properties of lubricating grease (Four-Ball Method). ASTM Int. 2002.
- [16] ASTM-D 2266, Standard Test method for wear preventive characteristics of lubricating grease (Four-Ball Method). ASTM Int., 2002.
- [17] K.R. Ranjit, Design of experiment using the Taguchi approach, New York: John Wiley & Sons Inc., 2001.
- [18] P.J. Ross, Taguchi techniques for quality engineering. Singapore: McGraw-Hill Int. Edition, 1996.