

# The Effect of Cyclic Speed on the Wear Properties of Molybdenum Disulfide Greases under Extreme Pressure Loading Using 4 Balls Wear Tests

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**Abstract**—The relationship between different types of Molybdenum disulfide greases under extreme pressure loading and different speed situations have been studied using Design of Experiment (DOE) under 1200rpm steady state rotational speed and cyclic frequencies between 2400 and 1200rpm using a Plint machine software to set up the different rotational speed situations.

Research described here is aimed at providing good friction and wear performance while optimizing cyclic frequencies and MoS<sub>2</sub> concentration due to the recent concern about grease behavior in extreme pressure applications. Extreme load of 785 Newton was used in conjunction with different cyclic frequencies (2400rpm -3.75min, 1200rpm -7.5min, 2400rpm -3.75min, 1200rpm -7.5min), to examine lithium based grease with and without MoS<sub>2</sub> for equal number of revolutions, and a total run of 36000 revolutions; then compared to 1200rpm steady speed for the same total number of revolutions. 4 Ball wear tester was utilized to run large number of experiments randomly selected by the DOE software. The grease was combined with fine grade MoS<sub>2</sub> or technical grade then heated to 750C and the wear scar width was collected at the end of each test. DOE model validation results verify that the data were very significant and can be applied to a wide range of extreme pressure applications. Based on simulation results and Scanning Electron images (SEM), it has been found that wear was largely dependent on the cyclic frequency condition. It is believed that technical grade MoS<sub>2</sub> greases under faster cyclic speeds perform better and provides antiwear film that can resist extreme pressure loadings. Figures showed reduced wear scars width and improved frictional values.

**Keywords**—MoS<sub>2</sub> grease, wear, friction, extreme load, cyclic frequencies, aircraft grade bearing.

## I. INTRODUCTION

THE use of MoS<sub>2</sub> greases are most useful in the boundary lubrication regime where metal to metal contact exists, in contrast to the hydrodynamic regime where a sufficiently thick fluid film exists to prevent asperity contact and where virtually no wear takes place. Greases are classified as shear thinning or pseudo-plastic fluid, which means that the viscosity of the fluid is reduced under shear. After sufficient shear force, the viscosity drops and approaches that of the base lubricant, such as mineral oil. Generally molybdenum disulfide in its naturally occurring hexagonal form is chemically very inert. It is insoluble in both oil and water. The technical and fine grade MoS<sub>2</sub> additives in greases have been in use over the years to achieve antiwear and load bearing capacity; they have varying

degree of successes and are very important for extreme pressure situations [1]-[3]. Even after considerable degree of oxidation, molybdenum disulfide can still deliver an acceptable level of lubrication performance [4]. It has also been cited as a useful additive in titanium complex greases that in themselves have inherently high load carrying capability, to obtain even better performance [5].

In previous studies different rotational speeds and extreme pressure loading were conducted by Nehme et al. on fully formulated oils and plain ZDDP oils [6]-[9]. It has been found that varying sliding speed and contact load will affect tribofilm formation and additives interactions. A substantial effort was devoted to understand these interactions by using design of experiment software (DOE) model.

This research examined different MoS<sub>2</sub> grades in lithium based grease under extreme pressure and different speed testing using chrome plated steel balls (Bearing-quality) which simulate the conditions of high pressure contact in real applications. The balls are aircraft grade E52100. During landings, the aircraft bearings are subjected to shock loads with very rapid acceleration. Most bearings should be protected from excessive wear and heat by certain form of grease or lubricant. This research shows that at accelerating frequency and speed, the use of technical grade molybdenum disulfide greases will enhance the superior resistance to scuffing and scoring under an extreme load of 785 Newton as indicated by the wear scar data presented in Table II. Nehme and his team worked extensively on previous tests using different Design of Experiment combinations to check the performance of the grease lubricant with respect to different speeds and steady state speed [10]. Different rotational speeds were studied previously on several oil combinations and proved effective in preventing wear depending on the process and applications [11]-[14].

The idea of MoS<sub>2</sub> assisted lubrication has been proposed as a promising concept. None the less, despite the numerous types of nanoparticles that have already been tested and some that have already been used in practical applications, the unquestionable and precise tribological mechanisms, as well as the key influential parameters under different contact conditions have yet to be determined [16]. Therefore, the Design of Experiment approach to understand the interaction of MoS<sub>2</sub> particles in lithium base grease and the investigation into the Effect of Molybdenum Disulfide and Graphite on the Load Bearing Capacity of Greases carried out by previous

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researchers indicated great improvement under extreme loading conditions [2], [15], [16].

## II. EXPERIMENTAL PROCEDURE

Lithium based grease containing 3% of MoS<sub>2</sub> were prepared in two batches of 100 grams; one batch with technical fine grade MoS<sub>2</sub> (particle size in the order of 5 $\mu$ m), and another batch with fine grade MoS<sub>2</sub> (particle size in the order of 2 $\mu$ m). The sequence of adding MoS<sub>2</sub> to the grease and mixing it is important for the final preparation. The mixture was mechanically mixed in the blender for 1hour. Three compositions of this grease with 3% MoS<sub>2</sub> and without MoS<sub>2</sub> were developed.

A Plint Four-ball wear tester (Model number TE92, ASTM standard D2266) was used to run these tests at the University of Texas at Arlington laboratory. Four chromium steel balls with bearing quality and aircraft grade E52100 were used. Three in number, of 1/2 inch diameter were clamped together and covered with grease, and the fourth was clamped in a ball chuck and the load was applied. The temperature of the grease was maintained at 75°C. A program was written for each specific condition to adjust for the speed, load, and the cyclic conditions and is presented in Table I. Coefficient of friction and the surface temperature of the cylinder as a function of the number of revolutions or time duration were measured directly and an excel file was generated.

Post-test analysis such as wear scar width of the rolling and fixed balls was measured and examined using a JEOL JSM 845 Scanning Electron Microscopy (SEM) at the end of each test providing that the test balls are cleaned with hexane-acetone mixture to remove the debris and grease from the surface. The average wear width was calculated and inputted in the design of experiment matrix for analysis. Evaluation of the factors and responses base on the percentage probability vs. effects and the ANOVA were investigated. The optimized conditions were calculated using desirability value.

## III. RESULTS AND DISCUSSION

### A. Tribological Results of Greases with MoS<sub>2</sub> Additives

Several tests with different grease blends were used to investigate the frictional events and wear under different speeds and an extreme load of 785N. Fig. 1 (a) depicts the typical progression of friction when small particles fine grade MoS<sub>2</sub> was tested under a steady speed of 1200rpm. The rise in friction can be easily identified and it might be due to the breakdown of the protective antiwear film which corresponds to the abrasive action of the debris present in the wear track as shown in scanning electron image. The stable antiwear film formed in the cyclic frequency speed at 785N extreme load is responsible for the low friction coefficient when using both technical and fine grade MoS<sub>2</sub>. The dominance of the beneficial effects of the tribofilm for protection of the surface is very significant and it is reflected in the SEM images of Fig. 1 (a). It is important to note that technical grade MoS<sub>2</sub> performed relatively better under the same tribological conditions and the frictional data with the SEM image support

this finding. Very small particles can potentially generate some corrosion issues due to the higher acid numbers. In general, mid size particles with median particle size ~ 5 $\mu$ m (corresponding to Climax Technical Fine Grade) is the most commonly used MoS<sub>2</sub> particle size in greases [5].

Wear scar width for several tests were part of Design of Experiment software (Table II) to analyze several factors related to speed and MoS<sub>2</sub> concentration. A two level factorial design with 2 replicates (test repeated two times) was set to identify the variables to be studied and their ranges. The set up analyzed the measured outcomes that clearly showed the importance of technical grade MoS<sub>2</sub> greases when used in repeated frequency applications such as extreme loads with very rapid acceleration.

Fig. 1 (b) represents the frictional events and the wear tracks for selected tribological test samples conducted with technical grade MoS<sub>2</sub> greases under cyclic frequency and steady speeds. Tests were repeated two times for the same grease blend and showed a great consistency with error less than 10%, which is insignificant considering the number of deterministic and non-deterministic variables in a tribological test. The break down region of the tribofilm showed a rapid increase in friction coefficient and abrasive wear resulted under steady speed of 1200rpm. The abrasive wear and the protective antiwear film depend greatly on the cyclic speeds under an extreme load of 785 N. On the other hand, the wear scar width variation was very clear under an extreme load of 785 N without MoS<sub>2</sub> additives (Table II). Therefore greases with no MoS<sub>2</sub> of any kind results in significant increases in wear scars and were exclude from the final optimization analysis.

### B. Optimization of MoS<sub>2</sub> Additives with Respect to Different Speeds

After calculating the average wear scar width on the three stationary balls that is presented in Table II, a DOE optimization process was conducted for the three factors considered in the experiments. The DOE focused on the desirability value and its significance during analyses. Desirability is used when multi objective optimization is sought according to Barrentine [17]. According to this approach, all factors and responses used in the analyses should be converted into corresponding desirability functions. The desirability is high when response approaches its target value. The desired goal is minimum wear scar for optimum MoS<sub>2</sub> concentration. Fig. 2 (a) indicated that cyclic frequency performed better when compared to 1200rpm under the same loading conditions. Moreover, technical grade molybdenum disulfide in grease can be reduced to approximately 2.25% when using cyclic speed without changing the outcomes, since the desirability is very significant at that concentration. On the other hand, the performance of technical grade MoS<sub>2</sub> with lower concentration under steady speed is diminished due to the higher wear scar width and the lower desirability (Fig. 2 (b)). The analyzed DOE data corroborated closely with the frictional events and the previous SEM images.

Two optimized samples of 2.25% technical grade MoS<sub>2</sub> grease were tested under an extreme load of 785 Newton using cyclic speed and steady speed. Frictional events were measured and high magnification SEM images were presented. The difference in both optimized samples was very significant, which demonstrated increased wear at the steady state speed (Fig. 2 (c)). It can be concluded that both samples measured experimental data corroborated closely with the Design of Experiment optimization.

#### IV. CONCLUSION

MoS<sub>2</sub> was used as EP additive and it has shown great improvement over the greases without MoS<sub>2</sub> (Table II). MoS<sub>2</sub> greases between 2.25 and 3% exhibited great reduction in the wear width under cyclic speed when compared to 1200rpm

speed especially technical grade was used. The presence of MoS<sub>2</sub> in grease under cyclic speed improved the wear condition in the four balls wear test. SEM images showed clearly the outperformance of technical grade MoS<sub>2</sub> in grease. DOE method was used to compare the wear performance and results clearly indicated the superior load bearing capacity of the MoS<sub>2</sub> greases when tested at cyclic frequency speed under extreme loading conditions. The technical grade optimized condition was considered for fundamental analysis using Scanning Electron Microscopy (SEM) and it was found that large abrasive particles existed in the wear tracks at the 1200 rpm tests when compared to the smooth wear tracks of the cyclic speed tests. Therefore, the improvement under cyclic speed can be critical for molybdenum disulfide grease under high pressure applications.

TABLE I  
PROGRAM SET UP ADJUSTMENTS USING PLINT MACHINE SOFTWARE (TE92) FOR VARIOUS TESTING PROCEDURES

Comment	Step No.	Next step	Loop count	Step time	Data mode	Motor enable	Load Newton	Specimen Temperature	Clutch Enable	Counter Reset	Speed rpm	Drive enable	Test Control unit	
Apply Load	1	2	0	CStp+		Disabled	785	20	Disengaged	Reset/on	0	Enabled	Current time	
Wait for 75 C	2	3	0	CStp+		Disabled	OR	75	Disengaged		0	Enabled	Elapsed time	Per test
Reset PID	3	4	0	2		Disabled	OR	OR	Engaged		0	Enabled	Current step	
Run Test	4	5	0	225	10 sec	Enable	OR	OR	Engaged		2400	Enabled	Total time	Per step
Decrease rpm	5	6	0	CStp+		Enable	OR	OR	Engaged		1200	Enabled	Elapsed time	Per step
Reset PID	6	7	0	2		Enable	OR	OR	Engaged		1200	Enabled	Residual time	Per step
Run Test	7	8	0	450	10 sec	Enable	OR	OR	Engaged		1200	Enabled	Test status	
Increase rpm	8	9	0	CStp+		Enable	OR	OR	Engaged		2400	Enabled	Test file	
Reset PID	9	10	0	2		Enable	OR	OR	Engaged		2400	Enabled	Data file	
Run Test	10	11	0	225	10 sec	Enable	OR	OR	Engaged		2400	Enabled	Data points	In step
Decrease rpm	11	12	0	CStp+		Enable	OR	OR	Engaged		1200	Enabled	Total data	
Reset PID	12	13	0	2		Enable	OR	OR	Engaged		1200	Enabled		
Run Test	13	14	0	450	10 sec	Enable	OR	OR	Engaged		1200	Enabled	start	
Stop motor	14	15	0	10		Disabled	OR	0	Disengaged		0	Enabled	stop	

TABLE II  
DESIGN OF EXPERIMENT (DOE) DATA FOR ANALYSIS THAT REPRESENT SEVERAL VARIABLES

Standard	Run	Factor1: Technical grade vs. fine grade	Factor2: MoS <sub>2</sub> % concentration	Factor3: Cyclic speed vs. steady speed	Response: Wear scar width(mm)
3	1	Technical grade	0	Cyclic speed	1.33
15	2	Technical grade	3	steady speed	1.15
10	3	fine grade	0	steady speed	1.29
12	4	Technical grade	0	steady speed	1.35
6	5	fine grade	3	Cyclic speed	0.90
16	6	Technical grade	3	steady speed	1.24
9	7	fine grade	0	steady speed	1.19
4	8	Technical grade	0	Cyclic speed	1.31
11	9	Technical grade	0	steady speed	1.34
7	10	Technical grade	3	Cyclic speed	0.73
14	11	fine grade	3	steady speed	0.91
13	12	fine grade	3	steady speed	0.88
8	13	Technical grade	3	Cyclic speed	0.70
2	14	fine grade	0	Cyclic speed	1.24
1	15	fine grade	0	Cyclic speed	1.22
5	16	fine grade	3	Cyclic speed	0.86

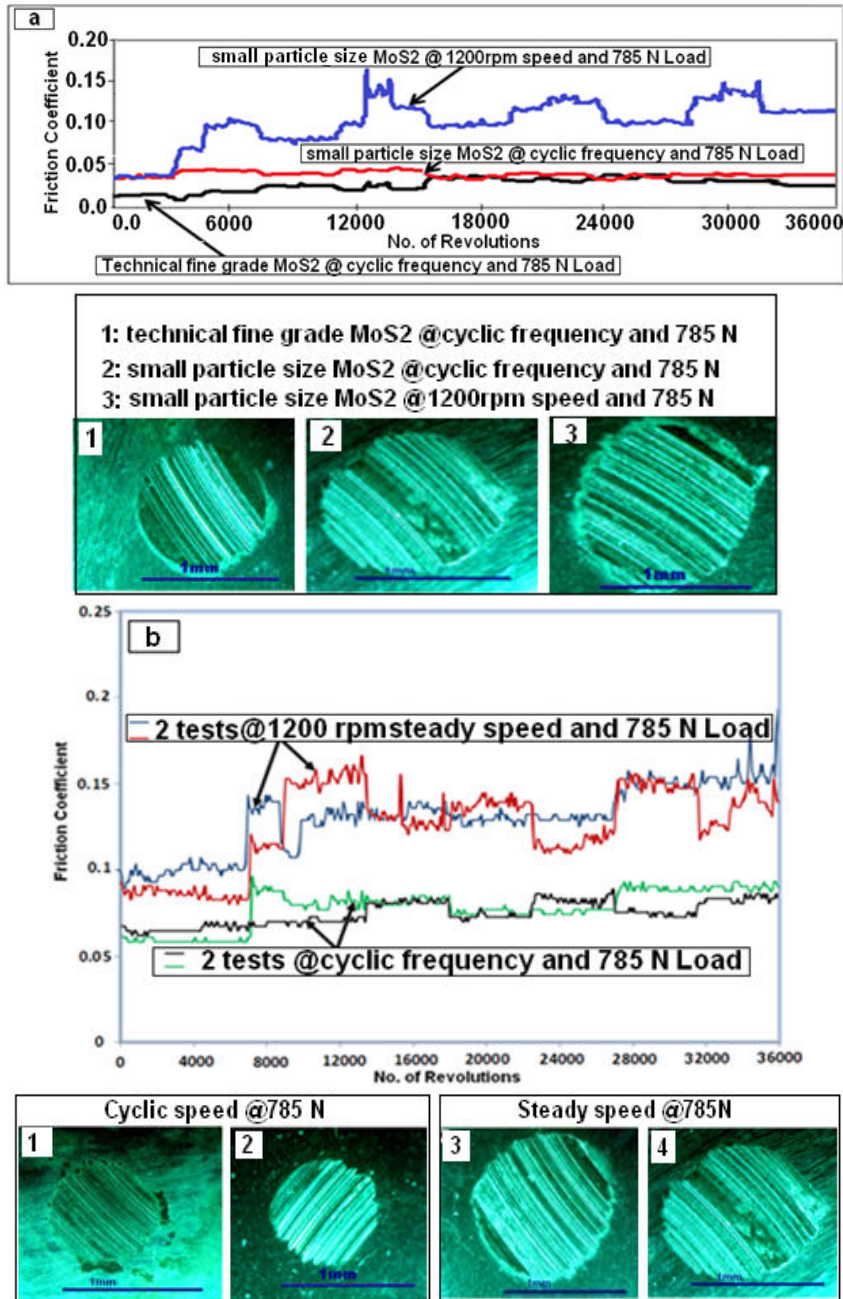


Fig. 1 Lithium based grease with 3% MoS<sub>2</sub> under extreme pressure loading: (a) represents frictional events and SEM images of technical fine grade and small particle fine grade MoS<sub>2</sub> greases @ cyclic speeds and 1200rpm speed for 36000 revolutions; (b) represents frictional events and SEM images of two repeated tests for technical fine grade MoS<sub>2</sub> greases to check for consistency

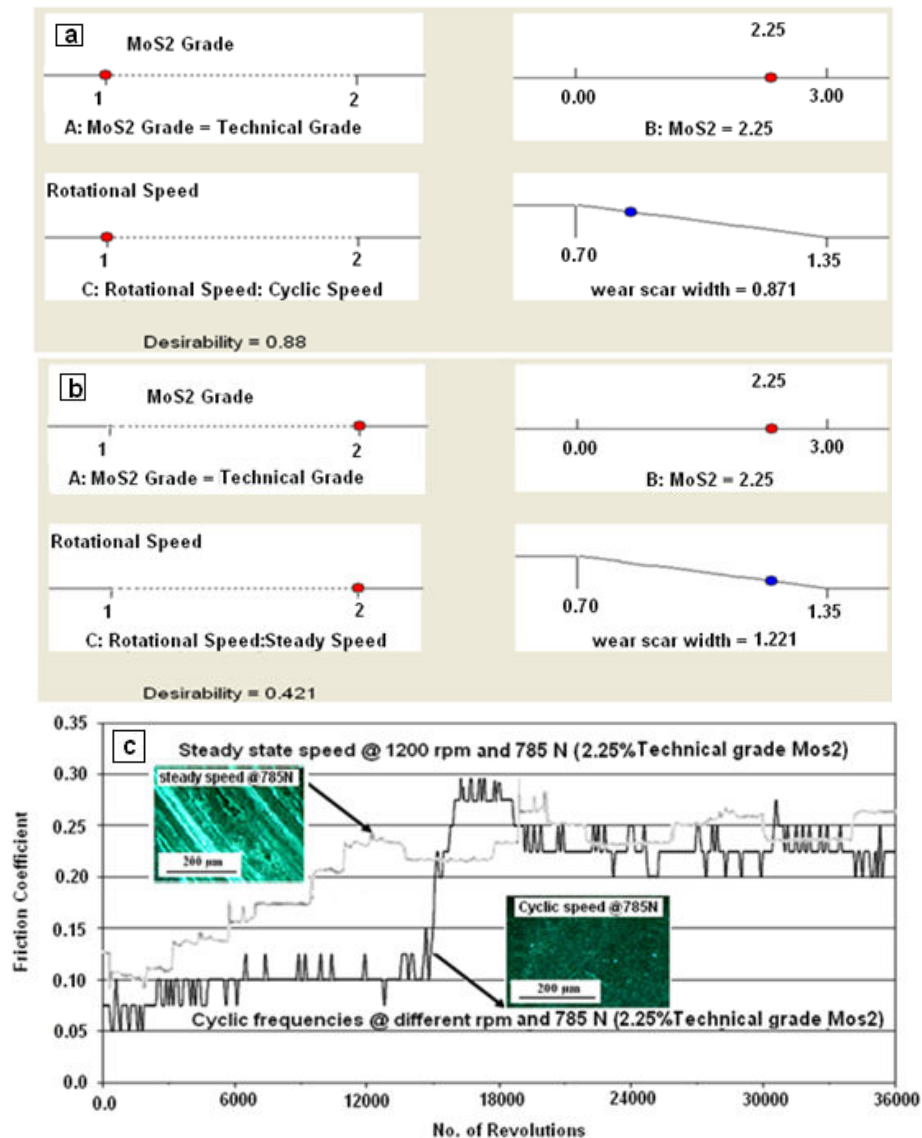


Fig. 2 Lithium based grease with an optimized 2.25% technical grade MoS<sub>2</sub> under extreme pressure loading: (a) and (b) represent the DOE optimization process; (c) represents the frictional events with high magnification SEM images of the optimized conditions

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