ISSN 1392-1134/ eISSN 2345-0371
Agricultural Engineering, Research Papers, 2019, Vol. 51, 1 – 7

EFFECTS OF BREAK IN PERIOD ON THE 4-BALL WEAR TESTS USING MOLYBDENUM DISULPHIDE (MOS_2) AS EP ADDITIVES IN LITHIUM BASED GREASE

Gabi Nehme*, Sara El-Merhabi*, Saeed Ghalambor**

* Department of Mechanical Engineering, University of Balamand, Lebanon
** Department of Mechanical Engineering, University of Pittsburgh, USA

Abstract: The lamellar structure of MoS_2 provides very good protecting layer that can be easily sheared under the application of extreme pressures and thus enhance the antiwear performance of the chromium steel bearing aircraft grade E52100. ASTM standard 2266 uses 4–ball testing by keeping the load at 393 Newton wherein the grease is heated to 75ºC and the test is conducted at a rotational speed of 1200 min^{-1} for 1 hour and the wear scar is measured at the end of the period. While this method is useful under fixed conditions it is not always representative of the fields where variable loads and speeds and different break in periods play a role and could change the results tremendously.

The goal of this study was to examine the loads and speeds conditions with and without break in period of 30 second at the start of the test and every 7.5 minutes thereafter. This will help explain their influences on the wear properties of lithium base grease under fixed 2% MoS_2 concentrations. Results indicated that wear was largely dependent on the loading condition during specific break in period at a higher rotational speed. It is believed that MoS_2 greases perform better under moderate loading when break in period is used as was optimized by Design Expert analysis (DOE). MoS_2 is sufficient to improve the wear characteristics at different break in period for different speeds and loads for extreme pressure applications.
Keywords: Friction, Wear, Molybdenum disulfide, grease, Extreme pressure, load with break in period, Design of Experiment (DOE).

Received 2019-10-12, accepted 2019-12-05

Introduction

Grease lubricants have great advantages. They can be used in harsh environment and have long life without contamination when compared to oils. Grease is a semifluid to solid mixture of a fluid lubricant, a thickener, and additives. It performs like mineral or synthetic oil in the lubrication regime but with the aid of thickener such as soap, or organic and inorganic non-soap, its consistency will be enhanced and it will perform better in high-pressure applications [1]. The majority of greases on the market are composed of mineral oil blended with a soap thickener.

It is reported that MoS2 particles mixed in oil, grease or impregnated into porous matrix of powdered materials appear to enhance the tribological properties in definite loading range in comparison to typical oil additives [2, 3]. Grease are mixtures of fluid lubricant (petroleum oil, synthetic or vegetable oils), thickener (soaps, organic or inorganic non-soap thickeners), and additives to enhance the performance and protect the grease and lubricant surfaces. After sufficient shear force, the viscosity drops and approaches that of the base lubricant, such as mineral oil. The additives in greases have been in use over the years to achieve anti-war and load bearing capacity; they have varying degree of successes. The chief among them are MoS_2 and graphite [4–6] for extreme pressure applications.

In previous studies, several loading and speed combinations were investigated using grease and oil lubricants with different type of additives [7–9]. It has been found that varying sliding speed and contact load will affect tribofilm formation and additives interactions. It was also shown that cyclic loading by ramping up or down will perform better than constant loading under higher min^{-1} speeds. A substantial effort was devoted to understand these interactions by using design of experiment software (DOE) model. The model was used to optimize several factors and responses for specific ranges of loads and rotational speeds. Design of Experiment reduces experimentation and increase the robustness of the testing procedure. Properly designed experiments and variables can yield an optimized product with very few tests. In addition, DOE provides significant ANOVA tables that can shorten the development cycle for product understanding and make optimization highly desirable.

In this study there are three important factors with two variables each (Table 1) that were examined to evaluate the potential influence of friction and wear on 52100 steel bearing (aircraft grade) immersed in MoS_2 lithium based grease. The variables studied are extreme pressure loading (40–80 kg), break in period (0 – 30 s), and rotational speed (600–1200 min^{-1})]. The two level factorial DOE which requires 2^3 experiments i.e. 8 experiments in full factorial design was used. The range of variables is usually set numerically or categorically at the low and high of the range where the low is taken as – by the DOE and the high is taken as + depending on the analysis that will be carried out after experimentations. The choice of variables and their ranges is a critical factor in developing a good factorial design and eventual development of an optimized product as was used previously in several tribology articles by Nehme et. al [6–11]. The DOE will provide insight on how the interactions of the variable influence the outcome of the tests by randomized the experiments to minimize the effect of external variables such as operator error, test environment, machine variability among others and reduce the uncertainty in the outcome of the model. Once the important variables are identified using the half-normal probability plots and the analysis of variance table (ANOVA), the interaction between these variables are determined and optimized using the desirability function.

Table 1. Design of Experiment (DOE) data for analysis of wear scar diameters after considering several factors with their variables.

Test No	Factor 2-rotational speed: 600–1200 min^{-1}	Factor 3-Break in period (s): 0–30	Factor 1– Load Range: 392–784 N	Response-Wear scar diameter (mm)
1	600	0	40	1.4
2	600	30	40	1.45
3	1200	0	40	1.35
4	1200	30	40	0.45
5	600	0	80	1.41
6	600	30	80	1.42
7	1200	0	80	1.46
8	1200	30	80	0.71

The goal of this research was to examine these variables, compare their friction, and wear characteristics to constant loading without a break in period. We deviated from ASTM D2266 standard to predict the wear behaviour by adding a break in period of 30 seconds. The grease was heated to 75°C, the test was run using several DOE conditions, and the wear scar was measured. Two compositions of this grease with 2% MoS_2 and without MoS_2 were developed to address the importance of cyclic frequencies applications. This research shows that higher speed with break in period using molybdenum disulphide greases will enhance the superior resistance to scuffing and scoring. Aerospace ball bearings are not always easily accessible for routine maintenance, but must perform flawlessly in varieties of environment. Therefore, break in periods and different speeds were used in these experiments to check the importance of molybdenum disulphide greases in preventing wear under EP conditions. Previous research without a break in period show different results as presented in different published articles [12–15].

Experimental Procedure

Greases were prepared in weight percent in Batches of 150 grams grease mixed with 2% MoS_2 were prepared in a Kitchen Aid blender (4.5-quart capacity with power rating of 250 watts). The sequence of adding MoS_2 to the grease and mixing it is important for the final preparation since lithium-based greases have higher melting point. Various different greases were evaluated and the typical sequence of additive introduction in the blending process started with base grease, to this was added the MoS_2 and stirred for five minutes, then the mixture was mechanically mixed in the blender for 30 minutes. Plint Four-ball wear tester (Model number TE92) was used to carry out the tests. The tested chromium steel balls were aircraft grade E52100 of ½–inch diameter. Three of them were clamped together and covered with grease, and the fourth was clamped in a ball chuck and the load was applied when the temperature of the grease reached 75^{o}C. A program was written for each specific condition shown in the DOE Table. The Plint machine software provides the setup of the program where the operator can adjust the components and variables base on the test conditions. The acquired friction data were plotted in excel and the wear scar diameters of the balls were measured by the scanning electron microscope (SEM) then averaged out and inserted in the DOE Table to be analyzed and optimized for all conditions. The software setup is easily adjusted and synchronized with various machine components.

Design Expert study the factors simultaneously and minimize experimentation, which is in direct contrast to the typical one-factor-at-a-time (OFAT), which limits the understanding and wastes data. The optimized conditions were concluded base on the effects, factors interactions and the analysis of variance: ANOVA. As stated earlier coefficient of friction as a function of number of revolutions or time duration were measured directly by the software and plotted using excel by subtracting the data of break in period since the machine stopped for 30 seconds every 7.5 minutes into the test and there were no data. In addition, at the start of the test with break in, we used a running in period of 30 seconds then we stop for 30 seconds then we run the test. Post-test analysis such as wear scar width was examined using a JEOL JSM 845 Scanning Electron Microscopy (SEM) at the end of each test providing that the test balls were cleaned with hexane-acetone mixture to remove the debris and grease from the surface.

Results and Discussion

Frictional Events And Wear Scars Analysis. Results indicated that friction and wear were largely dependent on the break in period at higher speed under 392 N loading. It is believed that MoS_2 greases under 1200 min^{-1} performed better (Table 1). An optimized condition of (1200 min^{-1}: 7.5 min + break in for 30 seconds for a total run of 36000 revolutions), under extreme loads of 40 and 80 kg showed reduced wear scars width and improved frictional values when compared to 600 min^{-1} under the same conditions (Figures 1–3).

Figures 1–2 present the consistency of two repeated tests at different contact load with break in periods compared to constant loads without break in at 1200 min^{-1} speed. It shows their friction coefficients as a function of number of revolutions. The stable anti-war film formed at 40 kg load was more apparent than at 80 kg load. The dominance of the beneficial effects of the tribofilm for protection of the surface is very significant and is reflected in the SEM images of Figure 1. The rise in friction can be easily identified at the beginning of the test under both loads and it might be due to the breakdown of the protective anti-war film, which corresponds to the abrasive action of the debris present in the wear track (Figure 1). The abrasive wear and the protective anti-war film depended greatly on the speeds, break in, and the extreme loads used. The variations in the repeated tests shown in Figures 1, 2 and 3 were less than 10%, which are insignificant considering the number of deterministic and non-deterministic variables in a tribological test. The average diameters of the wear scars on the balls was used to optimize the data using DOE analysis.

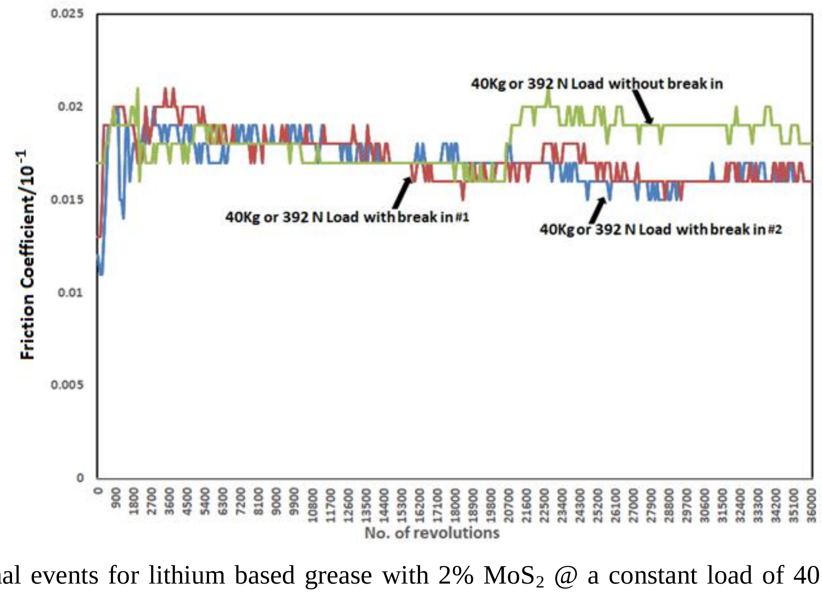

Figure 1. Frictional events for lithium based grease with 2% MoS_2 @ a constant load of 40 kg or 392 N and 1200 min^{-1} speed with and without break in period for 36.000 revolutions total.

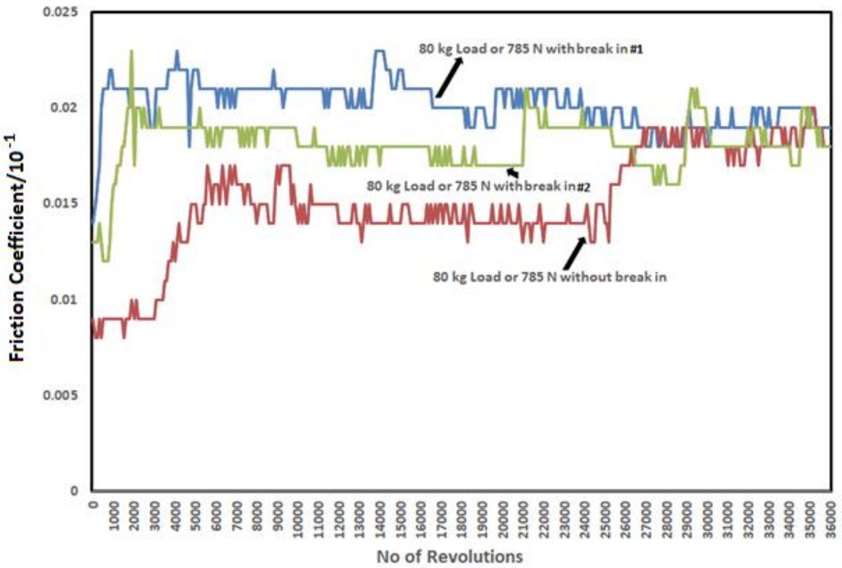

Figure 2. Frictional events for lithium based grease with 2% MoS_2 @ a constant load of 80 kg or 785 N and 1200 min^{-1} speed with and without break in period for 36000 revolutions total.

The large wear scars diameters for the 600 min^{-1} tests and the no break in experiments indicated very high degree of consistency as shown in Table 1. Therefore, the Design of Experiment analysis would be very significant when running optimization on these samples. 1200 min^{-1} speed shows significant decrease in wear at 392 N load with break in if compared to other tests and totally contradict previous articles when testing were used at low speed without break in [7–14]. The effect of MoS_2 grease at this speed was extremely beneficial when break in is used. . These scratches and the scattered wear debris can be clearly identified in the SEM images (Figure 3).

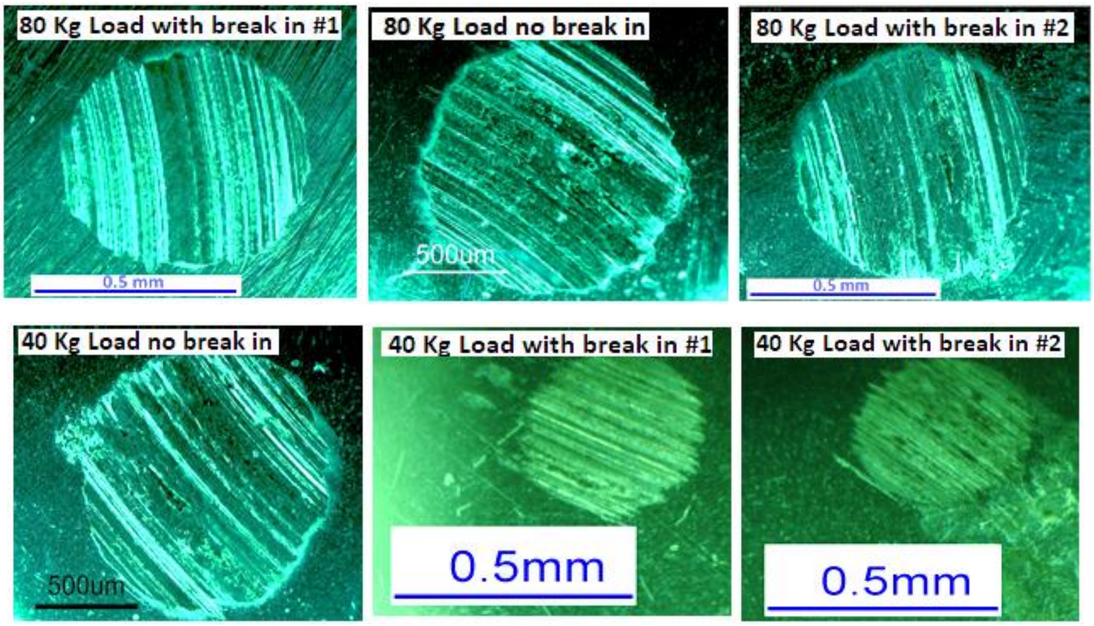

Figure 3. Scanning Electron Microscopy (SEM) of the wear scar diameters for Lithium based grease with 2% MoS_2 @ different loading conditions and 1200 min^{-1} with and without break in for 36.000 revolutions total.

Wear tracks were observed to be smoother and plateau region of stable friction was observed at toward the end of the experiment that lasted longer in the case of 40 kg load tests with break in when compared to the same events of the constant loading conditions for a total number of 36000 revolutions. On the other hand, some of the wear occurred when using 600 min^{-1} speed is abrasive and noticeable, since the tracks are very large and debris where there is abrasive pull out could be seen.

The protective molybdenum disulphide grease film breaks down was very limited in the case of 1200 min^{-1} with break in due to small amount of wear debris interaction with the tribofilm. As a result, some surface materials were removed and abrasive scratches were formed

Design of Experiment Analysis

Table 1 shows the wear scars data for the grease blends collected with and without break in periods under two different extreme loads (40, 80 kg) and different speeds in order to implement DOE analysis. Design of Experiment is the simultaneous study of several variables to establish desirability of the model base on the half normal probability and the ANOVA. DOE prediction of the wear scar diameter as a function of contact loads indicated large differences when a break in period is used and this can be clearly identified in Figures 4 and 5.

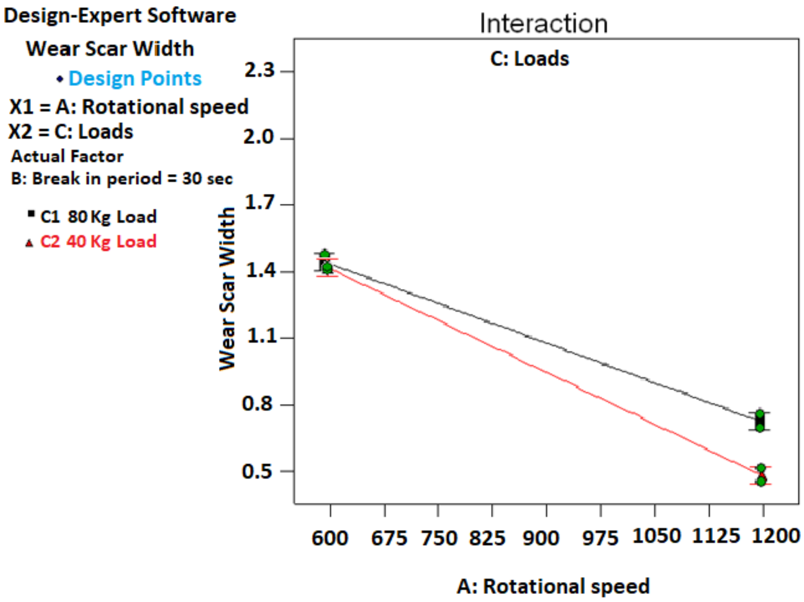

Figure 4. Design Expert analysis of the wear scar diameters when using different loads and speeds with a break in period.

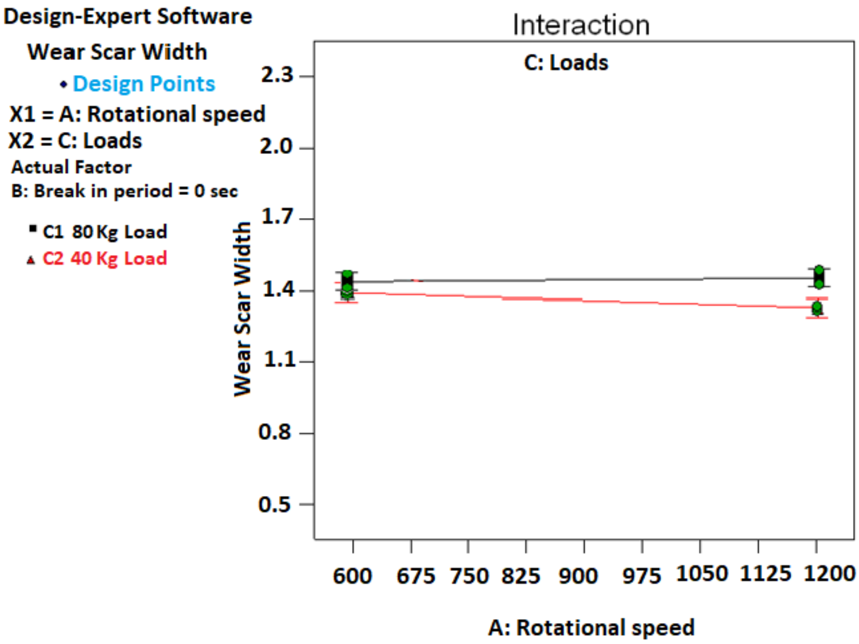

Figure 5. Design Expert analysis of the wear scar diameters when using different loads and speeds without a break in period.

The wear scar diameter was greater than 1.4 mm at lower speed with and without break in and at both loading conditions (Table 1). This will emphasize the usage of higher speed especially at the lower end of the loading conditions. These results are clearly supported by the wear width interaction figures and the SEM images for wear tracks. The SEM Figures show smooth surface with minimum amount of scratches at 1200 min^{-1} test with break in under the same loading condition. The DOE plots explore factors interactions for various wear width calculations. The central theme of these plots indicates that an optimum condition can be reached at 40 and 80 kg contact loads when speed is increased and break in period is used. Results indicated that MoS_2 grease is good extreme pressure lubricant and is responsible for reduced wear at higher frequency, break in period and extreme loading conditions.

Conclusion

In this study, a DOE method was used to compare the wear performance of greases with MoS_2 as EP additives under different contact loads and speeds using a break in period of 30 seconds every 7.5 minutes in the test. It is quite evident that the reduction of wear at higher frequency was the results of the break in period used during testing. Flat eventless regions of the friction coefficient associated with 1200 min^{-1} at 40 kg load and break in resulted in minimal wear whereas regions with large frictional events related to extreme load of 80 kg in increased wear even when break in is used. Wear scars data collected for tests conducted under extreme loads of 40 and 80 kg at lower speed indicated increase in wear with and without break in (Table 1). Statistical analysis is a powerful tool to predict the influence of individual variables on the desired outcome. Break in period played very important roles in the analysis resulting in reduced wear in several tests and enhanced the formation of antiwear film.

Acknowledgements

This research was supported by the University of Balamand at Lebanon. Testing Assistance provided by the lab staff at the University of Texas and Dr. Saeed Ghalambor is gratefully acknowledged.

References

[1] Gabi Nehme (2012), The Importance of extreme pressure cyclic load in Molybdenum Disulfide greases using 4 balls wear tests. In Proceeding of IJTC2012, STLE/ASME International joint Tribology Conference, Denver, Colorado, IJTC–2012.

[2] Gabi N. Nehme, The Tribological Performance of Plain and Fully Formulated Commercial Engine Oil under 2 Different Rotational Speeds and Extreme Pressure Contact Using Design of Experiment, Tribology Transactions, 54(4), 568–588.

[3] Gabi N. Nehme, The Effect of FeF_3/TiF_3 catalysts on the Thermal and Tribological performance of Plain Oil ZDDP under Extreme Pressure Loading. Wear, Volume 278–279 (2012): 1129–1147.

[4] Gabi Nehme, Interactions of fluorinated catalyst and polutetrafluoroethylene in two different plain zinc dialkyldithiophosphate oils and one fully formulated oil using design of experiment. Lubrication Science (2011), Volume 23, Issue 4, pages 181–20.

[5] Gabi Nehme, Performance Testing and Analysis of Anti–Wear Additives in Engine Oil for Reducing Phosphorus content and Improving Tail Pipe Emissions. University of Texas at Arlington, Ph.D Dissertation (2004).

[6] J. Gansheimer, R. Holinski Study of Solid Lubricants in Oils and Greases under Boundary Lubrication, Wear, 19(4), April 1972, pp. 439–339.

[7] A Misty, R. Bradbury. Investigation into the Effect of Molybdenum Disulphide and Graphite on the Load Bearing Capacity of Greases. NLGI Spokesman, **66(3)**, June 2002, pp. 25–29.

[8] A. Tamashuasky, The effect of Graphite type, purity and concentration on the Performance of a Clay Filled Polyalpha Olefin Grease Based on four ball wear (ASTM 2266) with coefficient of friction and load wear index (ASTM D2596). NLGI Spokesman, **65(12)**, March 2002, pp. 10–25.

[9] G. Nehme and M. Dib. Fluorinated Mix in plain ZDDP oil and Commercial Oil Using Design of Experiment analysis of all Interactions and Fundamental Study of Fluorinated Mix in plain ZDDP Oils under 2 different r/min Test Cycles and Extreme Boundary Lubrication, Proc. IMechE Vol. 225(4) Part J: J. Engineering Tribology, 193–201.

[10] P. Aswath, R. Mourhatch, K. Patel. S. Munot, A. Somayaji and R. Elsenbaumer, A Design of Experiments Approach to Develop a Better Grease, NLGI Spokesman, 71(4), 2007, pp. 8–16.

[11] Gabi Nehme (2011), Fluorinated FeF_3 catalyst interactions in three different oil formulations using design of experiment optimization and chemistry characterization of tribofilms. Lubrication Science 23 (4), 153–179.
[12] Gabi N. Nehme (2011), The Tribological Performance of Plain and Fully Formulated Commercial Engine Oil under 2 Different Rotational Speeds and Extreme Pressure Contact Using Design of Experiment, Tribology Transactions, 54(4), 568–588.
[13] Gabi Nehme (2013), The importance of variable speeds under extreme pressure loading in molybdenum disulfide greases using four-ball wear tests. Tribology Transactions 56 (6), 977–985.
[14] Gabi Nehme (2017), Tribological behavior and wear prediction of molybdenum disulfide grease lubricated rolling bearings under variable loads and speeds via experimental and statistical approach. Wear 376, 876–884.
[15] G Nehme. Tribological and thermal characteristics of reduced phosphorus plain ZDDP oil in the presence of PTFE/FeF_3/TiF_3 under optimized extreme loading condition and a break in period. Wear 301 (1–2), 747–752.

Author for contacts: Associate Professor: Gabi Nehme
E–mail: gabinehme@yahoo.com
or
gabi.nehme@balamand.edu.lb