

# INVESTIGATION INTO THE DYNAMIC PARTICLE GENERATION OF LUBRICATING GREASES USED IN SPACE MECHANISMS

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The purpose of this study is to examine the phenomenon of Dynamic Particle Generation in lubricating greases that are used in a variety of critical Aerospace mechanisms. Particle Generation occurs in bearings, ball screws, and other mechanical devices where dynamic conditions are present. This should not be confused with outgassing as particle generation is unrelated to the pressure effects on a system. This is a critical factor in many systems as particle generation can contaminate systems or processes causing them to fail. These failures can lead to excessive costs, production failure, and equipment damage.

In this study, several greases made from Multiplyalkylated Cyclopentane and Perfluoropolyether base fluids were tested to evaluate their particle generation properties. This particle generation phenomenon was studied using a custom test rig utilizing a high precision cleanroom ball-screw to simulate true application conditions. The ball-screw was tested at speeds from 200, 1,200, and 2,400 RPM to illustrate the effect of speed on the particle generation across different applications. This paper will show the tendencies of different lubricant chemistries to generate particles and which ones present advantages of improved durability and environmental cleanliness for critical processes and applications.

**KEY WORDS:** Particle Generation, Outgassing, Aerospace, MAC, Pennzane, Contamination, Perfluoropolyether, PFPE

## 1.0 Introduction

Contamination is an element of great concern in regards to mechanisms designed and planned for space flight. While there are many types of contamination including volatile outgassing, residual ions, airborne particulates, etc, the route that the contamination enters the system is just as important. Outgassing has been a well-documented method for contamination in a system and there are many ways to quantify it. Methods like ASTM E-595 and E-1559 that are used to generate control limits. The counterpart to outgassing or even the more typical volatile evaporation is called Dynamic Particle Generation

which can be as significant of a problem as outgassing. The term dynamic particle generation describes what happens when contaminants are created by being forced from a lubricated ball-screw, bearing, or gear system into the operating environment. These contaminants could include base oil constituents, thickener particles, additives, etc. all of which could be volatile or non-volatile.

The manner in which these contaminants are freed from the lubricant system is through dynamic mechanical action whether it be rolling, sliding, or a combination of both. This study will begin to investigate and illustrate that factors like mechanical and chemical compatibility, friction, speed, etc all have an effect on the amount of contamination generated through particle generation. I will also illustrate that physiochemical effects between lubricating fluids and thickeners play a key factor as well.

Aside from the space application environment, materials can also be contaminated through the manufacturing process even if done in a clean room environment. In these environments, the focus is typically on the quality of the air in the room and it has long been known that lubricants are a source of contamination in cleanroom and vacuum environments. Historically in order to alleviate the worries of many manufacturers, the solution over the last twenty years has been to ultrafilter<sup>1</sup> the lubricant to reduce the number of particles in the lubricant and the size of them.

There are three levels for cleanliness in a grease:

- Unfiltered grease – Can contain particles larger than 75  $\mu\text{m}$ .
- Filtered or so-called “Clean” grease – For example MIL-G-81322 Aircraft grease cannot have any particles greater than 75 $\mu\text{m}$  and there must be fewer than 1,000 particles/cm<sup>3</sup> between 24 $\mu\text{m}$  and 74 $\mu\text{m}$ .
- Ultrafiltered or “Ultraclean” grease – Such as MIL-G-81937 must not contain any particles greater than 35 $\mu\text{m}$ . In addition to this it cannot have more than 1,000 particles/cm<sup>3</sup> between 10 $\mu\text{m}$  and 34 $\mu\text{m}$  in size.

While these processes will certainly help remove or break up bulk and “hard” contaminants which will lead to smoother operation, reduced vibration, and lower noise in bearings, ball screws, and other motion applications, it may have little effect on the amount of particles “shed” or generated from a lubricant in a dynamic condition. So this leaves thoughts about what effect the lubricant truly has on this phenomenon and how does the base fluid, thickener, additive, and manufacturing processes effect this property and ultimately the application environment around it. The first step to investigate this new area of lubricant properties required the construction of a new test apparatus and creation of an accurate and repeatable test method [5].

### 1.1 Objective for Testing:

The primary purpose of this study was to use a newly developed test method and apparatus that could be used to accurately and repeatedly measure the Dynamic Particle Generation of a lubricating grease. This study centered around two different types of lubricants, Perfluoropolyethers (PFPE), and Multiply-alkylated cyclopentanes (MAC). MAC’s are composed of one cyclopentane ring with two to five alkyl groups substituted on the ring. The synthesis is performed by reacting dicyclopentadiene with various chain length alcohols producing a lubricant with a various range of physical properties [7]. PFPE’s are produced through the oxidation of hexafluoropropylene and are fully fluorinated oligomers that contain fluorocarbon links containing oxygen atoms. Two PFPE’s (Braycote® 601EF and NyeTorr® 6300) as well as three MAC lubricants (Rheolube® 2000, 2000F, and NyeTorr® 6200) were tested in this study

**Table 1:** Materials tested in study

	Braycote 601EF	NyeTorr 6300	Rheolube 2000	Rheolube 2000F	NyeTorr 6200
Base Chemistry	PFPE	PFPE	MAC	MAC	MAC
Thickener	PTFE	PTFE	Sodium	PTFE	PTFE

The Key Factors of this study will be.

1. To study the particle generation tendencies of different aerospace lubricants evaluated under dynamic conditions.

2. To examine the effect that speed plays on the particle generation of a particular system
3. To examine the profile of the particle generation results plot; that is how the system behaves with respect to time and number of particles generated.

### 2.0 Background:

To compare the dynamic particle generation characteristics of various lubricating greases, a custom test apparatus was designed and built to detect, analyze, and classify the products being examined. As this is a newly created test method and fixture, a Design of Experiments (DOE) was put together to investigate repeatability, variability, and statistical significance [5].

The core of the test apparatus is a high precision ball screw assembly meant for cleanroom and low outgassing applications. This ball screw design was used due to the fact that when lubricated correctly and under light load, it experiences virtually zero wear on its components. Since a ball screw assembly utilizes a system of rolling elements, the amount of frictional wear on any components especially under no load is greatly reduced. This allows us to study the particle generation solely based on the lubricant and speed of the test. With the addition of a lubricant that provides a protective film between all surfaces, the main component of wear on the system is the lubricating grease, which then generates the particles being examined in this study. This ensures that the particles generated in the dynamic system are purely generated from the lubricating grease. Please see Figures 1 through 4 for the Dynamic Particle Generation testing apparatus.

In Figure 1, the testing apparatus is illustrated showing the clean air supply that moves over the testing equipment, the ballscrew, and tunnel. To supply clean, filtered air to the system, a laminar flow clean air handler supplies clean ISO 2 Class air to the system. The air handler can deliver filtered air at a range of velocities, depending on operator input, but for the sake of repeatability and comparison, a velocity of 1m/s +/- .25 m/s was used for all testing. This value was decided on after investigating the average volume of clean air turned over in a clean air environment. This filtered air passes over the test system as the ball screw assembly operates for the length of the test.

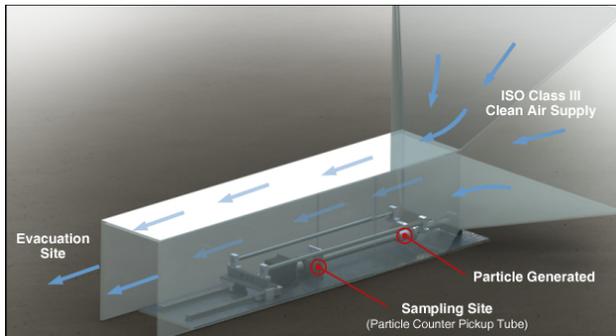


Figure 1. Dynamic Particle Generating test apparatus

Figures 2, 3, and 4 shows a more detailed view of the particle generation test unit and components.

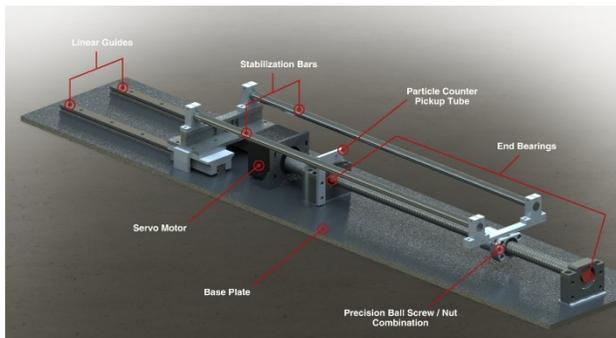


Figure 2. Dynamic Particle Generating test apparatus

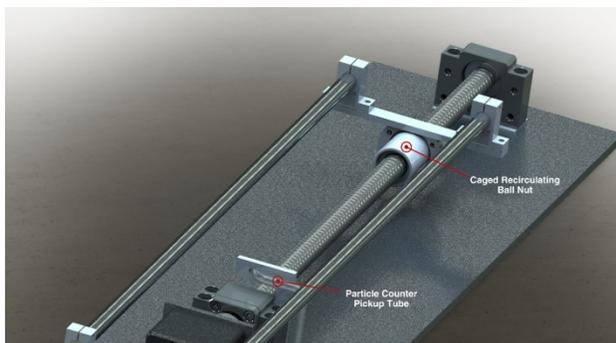


Figure 3. Dynamic Particle Generating test apparatus

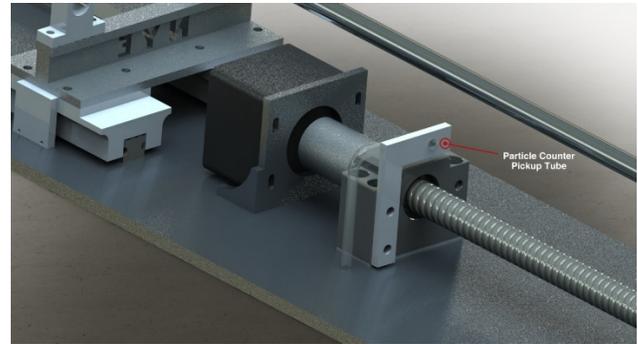


Figure 4. Dynamic Particle Generating test apparatus

Particles are collected via an inlet tube mounted at the end of the ball screw assembly (Figure 4.). The location of the pick-up tube is key, as it captures only particles generated by the grease on the ball screw, and none generated by the servo motor, bearings, linear guides, or flex coupling. The pickup tube leads to a Light-scattering Airborne Particle Counter. The particle counter features simultaneous particle measurement of sizes from  $0.1\mu\text{m}$  and above all the way to  $5\mu\text{m}$  and above via the use of a transverse light-scattering system which provides the most accurate and repeatable measurements available.

During testing a particle count profile is constructed, plotting the count of each category vs. test duration. This profile chart is an important asset to have in order to understand the behavior of the grease being run as two greases may share the same cleanliness value (ISO, JIS, or FED), but may have completely different particle distribution profiles over the duration of the test.

The dynamic particle generation test apparatus has the ability to run at speeds from 200 to 2,400 RPM but in this study, we looked at 200, 1,200, and 2,400 RPM respectively. This translates to 0.02, 0.1, and 0.21 m/s of linear velocity, respectively. Comparison between greases should only be conducted at like rotational speeds, as particle generation typically increases as RPM increases.

Grease is applied to the ball screw assembly at 200RPM with a sample volume of 2cc being applied via syringe and a 10 cycle run-in to evenly distribute the grease. The test begins with the motor stationary while the particle counter takes a series of background readings. The number of readings taken is user defined. The average of these background readings are subtracted from the particle count under dynamic conditions before ISO, JIS, or Federal Classes are

calculated. This ensures that the particles being counted are only those produced by the grease and not the filtered air passing over the system.

At the conclusion of the test, the collected data is compared against the particle classification tables and the ISO, Federal, and JIS classifications are determined. The ISO cleanliness levels are determined by the following formula and table.

$$C_n = 10^N \left[ \frac{0.1}{D} \right]^{2.08} \quad (1)$$

Where

C<sub>n</sub> = represents the maximum permitted concentration (in particle/m<sup>3</sup> of air) of airborne particles that are equal to or larger than the considered particle size; C<sub>n</sub> is rounded to the nearest whole number

N = ISO class number, which must be a multiple of 0.1 and be 9 or less

D = the particle size in microns

**Table 2:** ISO Particle Classifications in Air

CLASS	Number of Particles per Cubic Meter by Micrometer Size					
	0.1 micron	0.2 micron	0.3 micron	0.5 micron	1 micron	5 microns
ISO1	10	2				
ISO2	100	24	10	4		
ISO3	1,000	237	102	35	8	
ISO4	10,000	2,370	1,020	352	83	
ISO5	100,000	23,700	10,200	3,520	832	29
ISO6	1,000,000	237,000	102,000	35,200	8,320	293
ISO7				352,000	83,200	2,930
ISO8				3,520,000	832,000	29,300
ISO9				35,200,000	8,320,000	293,000

**Table 3:** Federal Standard Particle Classifications in Air

CLASS	MEASURED PARTICLE SIZE (MICROMETERS)				
	0.1	0.2	0.3	0.5	5
1	35	7.5	3	1	
10	350	75	30	10	
100		750	300	100	
1,000				1,000	7
10,000				10,000	70
100,000				100,000	700

## 2.1 Repeatability and Statistical Significance

To validate this new experimental test method, materials were tested with a minimum of three replications. Afterwards a statistical analysis of variance (ANOVA) was performed on the sample sets to look for any statistically significant differences. Different lots of the same material and different sections of a specific batch were also tested. The results of this statistical analysis can be found in the technical paper entitled “Investigation into the Dynamic Particle Generation of Lubricating Greases” [5].

## 3.0 Results and Discussion

Table 4 and 5 summarizes the ISO and Federal classifications for the five greases that were studied in this experiment at 200, 1,200, and 2,400 RPM respectively.

**Table 4:** ISO Classification of Aerospace Greases

	Braycote 601EF	NyeTorr 6300	Rheolube 2000	Rheolube 2000F	NyeTorr 6200
200 RPM	ISO 1	ISO 1	ISO 1	ISO 1	ISO 1
1200 RPM	ISO 4	ISO 4	ISO 4	ISO 2	ISO 4
2400 RPM	ISO 5	ISO 5	ISO 6	ISO 3	ISO 5

**Table 5:** Federal Classification of Aerospace Greases

	Braycote 601EF	NyeTorr 6300	Rheolube 2000	Rheolube 2000F	NyeTorr 6200
200 RPM	Class 1	Class 1	Class 1	Class 1	Class 1
1200 RPM	Class 100	Class 100	Class 100	Class 1	Class 100
2400 RPM	Class 1,000	Class 1,000	Class 10,000	Class 100	Class 1,000

It is apparent from Tables 4 and 5 that at the lowest rotational speeds, all of the lubricants performed equivalently. As the speeds increased as well as friction between the lubricants and the bearings, the materials started performing differently.

The two PFPE greases received identical ISO and Federal ratings so they appear to be equivalent from the perspective of how much particle contamination they generate into the environment. Looking at the particle generation graphs in Figures 5 and 6, a different story is told as the curves are dramatically different at 2,400 RPM. While both of these greases are PTFE thickened and made from similar viscosity linear structured PFPE fluids, their tendency to generate particles less than 1μ is very different. It is well known that PTFE and PFPE chemistries have weak

bonding strengths (Van Der Waals) between the molecules and surfaces during sliding motion [6].

However the difference seen here between these two materials is most likely the effect of the difference in PTFE used in the material. There are many different grades of PTFE as well as manufacturing processes ranging from emulsion formed polymers to irradiated polymers. These differences in manufacturing processes as well as residuals of surfactants and other chemical processing aids can possibly have a significant effect on the ability of the PFPE and PTFE to bind into a strong lubricant system. This combined with differences in the particle size distribution of the PTFE polymers creates factors that generate more environmental contamination through particle generation.

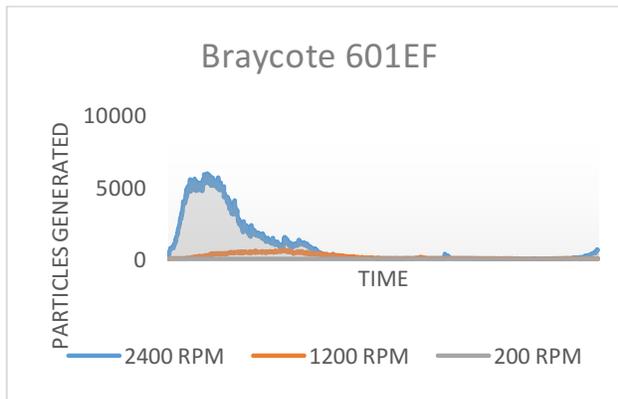


Figure 5. Dynamic Particle Generation of Braycote 601EF

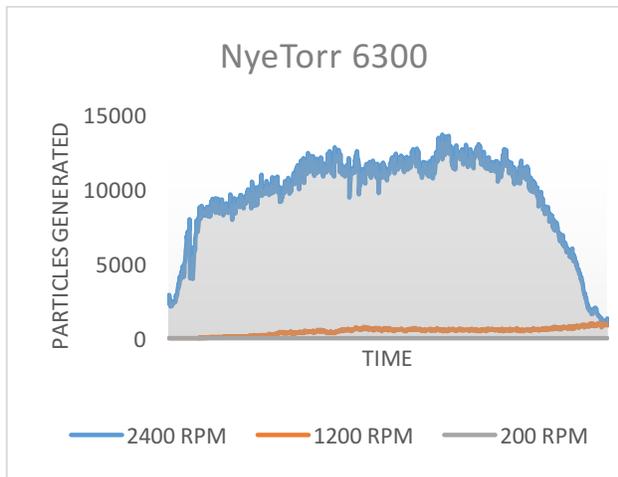


Figure 6. Dynamic Particle Generation of NyeTorr 6300

The results for the MAC based greases showed some potential performance benefits compared to the PFPE lubricants but it did depend greatly on thickener. The Rheolube 2000 and 2000F share the same base fluid while the NyeTorr 6200 utilizes the Pennzane Ultra X-2000 base fluid which is processed additionally to reduce its outgassing. The sodium thickener used in the 2000 grease is apparently sensitive to the shear as the material generates more particles as the rotational speed and sliding friction increases. In Figure 7, the particle generation trend can be seen to continually increase over time. The majority of these particles were small (less than  $1\mu$ ) and the continual increase in the amount detected per cubic cm of air is most likely attributed to the processing of the thickener/grease and the bonding strength of the sodium thickener resulting in the ultimate shear stability of the thickener.

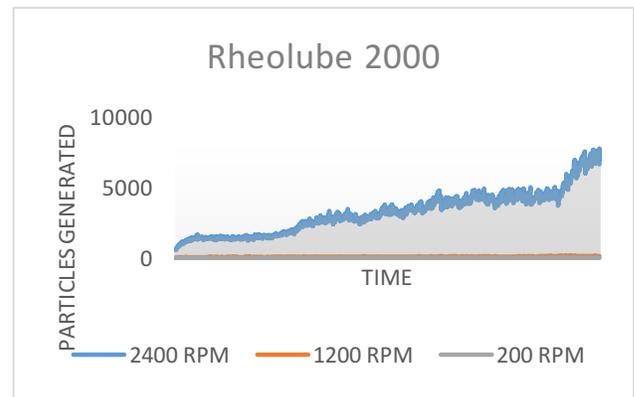


Figure 7. Dynamic Particle Generation of Rheolube 2000

As the 2000F and the 2000 grease both share the X2000 base oil and similar additive packages, we can see the direct effect of the thickener of the particle generation. It is well known that the MAC's possess superior film strength and adhesion to metal surfaces when compared to PFPE's so we would expect low particle generation as the probability of particles being shed to the air would be lower. With the thickener of the 2000F having strong covalent bonding within the PTFE molecules [6] combined with the strong film strength of the MAC, it showed to be the superior lubricant in this study for particle generation.

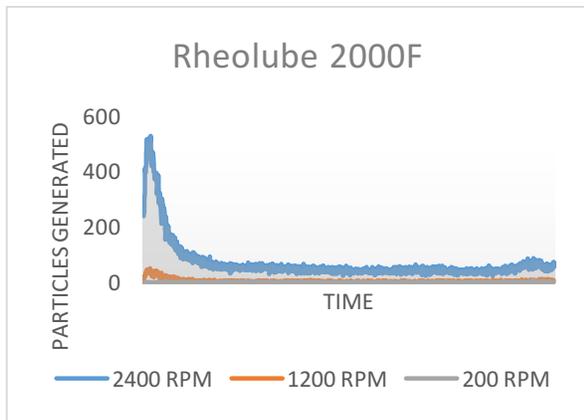


Figure 8. Dynamic Particle Generation of Rheolube 2000F

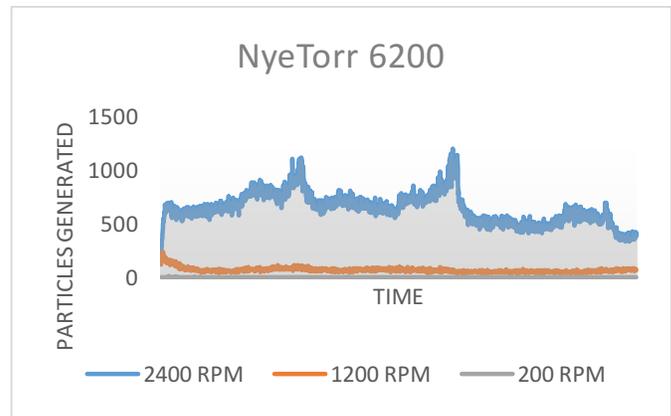


Figure 9. Dynamic Particle Generation of NyeTorr 6200

The results in Figure 9 for the NyeTorr 6200 which is made from the X-2000 Ultra (further refined MAC) and PTFE are very interesting as the amount of particle generation trends higher throughout the entire test than the 2000F and is classified one order of magnitude higher in both the 1,200 and 2,400 RPM tests. Beyond this difference, the 6200 generated ten times the amount of particles less than  $1\mu$  when compared to the 2000F. Although this is within the allowed error for the Federal Cleanliness standards, it is a significant difference when it comes to space critical applications. The issue is certainly not created by volatility as the X-2000 Ultra has lower ASTM E-595 results as well as Knudsen Vapor Pressures compared to the traditional X-2000 fluids. Since the manufacturing process, additives, and PTFE polymer type are all the same, this leads us to conclude there must be some effect from the X-2000 Ultra on the particle generation of the 6200.

While the X-2000 Ultra contains less volatiles and has demonstrated that it has superior outgassing properties to the standard grade, the particle generation is negatively affected. The reasons for this could be as simple as the refinement of the X-2000 has caused a change in the tribological properties of the fluid which has led increased friction between the fluid, PTFE polymer, and bearing surface. This additional friction could lead to degradation of the lubricant which then leads to shedding of particles from the lubricant system. Another possible cause for this particle generation could also be the removal of something from the X-2000 fluid that helped with the film strength or the compatibility with the PTFE polymer and its removal has promoted the increase in particle generation.

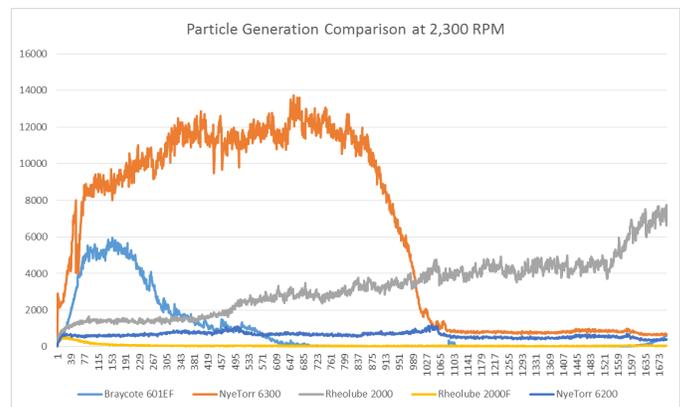


Figure 10. Comparison of the Dynamic Particle Generation

In figure 10, all of the samples tested in this study have been graphically plotted on the same axis to visually illustrate the differences in the particle generation between different materials. The 2000F clearly has the best performance of any of the materials tested.

#### 4.0 Conclusions

Determining the volume of contamination generated from a lubricant through particle generation in dynamic conditions is a new area just starting to be explored. As presented in this study, many factors play into the behavior of a grease in respect to particle generation characteristics. Variables such as run speed, base oil chemistry, and thickening agent

properties all play into how much contamination a lubricant will generate through particle generation.

This study has also shown that the 2000F which is a PTFE thickened MAC grease produced the least amount of contamination due to particle generation as well as the least amount of change in particle generation over speed. It was also illustrated that while MAC lubricants have an advantage over PFPE base ones, it certainly depends on other factors like the thickener, additives, processing, etc. This dependency was clearly seen in the sodium thickened Rheolube 2000. The organic soap thickener forms a strong matrix of entanglements but at higher speeds it generated more particles as a result of the shear stability of the sodium soap vs. the PTFE thickened greases. This is due to the fact that PTFE is a very stable molecule with Carbon-Fluorine bonds being one the strongest known. This combined with the higher RPM creating higher shear on the thickener at the metal surface causing more particles to generate over time. This leads to the conclusion that PTFE is the superior to sodium soap in particle generation and based on the differences in thickener stability under the shear conditions of the test.

The ability to plot particle generation over time and see differences in the distributions of various materials, will help us to form hypothesis about particle generation over time using normalized probability. Utilizing this type of analysis can also be used to predict lubricant service life. From these estimations, we could also look into ways to predict saturation by contamination in a space mechanism or the probability for success/failure when using a certain lubricant.

#### **4.1 Future Work**

The expansion of this work will include using Residual Gas Analysis (RGA) to determine the molecular weight and chemical species of all contamination materials being generated in the particle generation test. Further research will also be placed on the difference of PTFE polymers, X-2000 Ultra, manufacturing techniques, etc. and how this relates to bonding strength and in turn particle generation. We will also investigate the particle generation of a rolling element bearing system in atmospheric and vacuum conditions. The main goal of this work is to create a model and methodology to be able to predict the failure probability of a mechanism due to contamination when a certain lubricant is used.

#### **4.2 Acknowledgements**

I would like to thank my colleagues in the Application Development and Validation Testing (ADVT) Lab at Nye Lubricants Inc. who conducted the Dynamic Particle Generation testing, specifically Mason Wood. I would also like to thank William Galary for all of his support and consultation about the Clean Room Industry and its technologies. Finally I would like to thank Gus Flaherty for his work with me on designing and constructing the Dynamic Particle Generation testing equipment.

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