COMPANY OVERVIEW

Digital documents and references are made clickable for convenience, where appropriate.

Quick summaries of innovation are available in videos:

TriboTEX elevator pitch (1 min) **Singularity application (3 min)** **Kickstarter video (3 min)**

TriboTEX is introducing a consumer product line with one objective; dramatically reduce friction in both legacy and modern transportation vehicles. We bring a revolutionary approach to lubrication by synthesizing nanomaterials that promote self-organization to form low-friction boundary films when added to existing oils (both base and formulated). The result is a super-slick, silicon-rich, diamond-like carbon (DLC) coating on engine component interfaces and gear surfaces in gearboxes. The lubricious coatings form *in situ* (during operation) from our proprietary, flat, nano-sheets that are synthesized with functionally different sides.

TriboTEX developed synthetic nanotechnologies demonstrating SBIR Phase I feasibility for energy efficiency and longevity in existing machinery and transportation. Developments were guided by extensive bench testing and validated using vehicle tests under real world conditions. After considerable research and optimization, we have increased capacity for nanomaterial production under the SBIR Phase II program. Now we present to you the next big step in lubrication.

DEMONSTRATED PROPERTIES OF TRIBOTEX™ GENERATED COATINGS

- ✓ Formation of complex nanostructure coatings
    - Crystalline nanograins are intermixed with an amorphous matrix
- ✓ Plane alignment of nanocrystalline oxides/carbides correspond to typical "Superlattice" structures
    - Intergranular diffusion of magnesium into iron matrix slows down diffusion of oxygen
- ✓ Top layer consists of silica-doped DLC nanocomposite
    - Defines dynamic superlubricious properties
- ✓ Device level impact on decreasing fuel consumption
    - Up to 10% in larger, older vehicles (ex. 1989 GM K2500 5.7L V8, small block 350)
    - ~5% newer light weight and hybrid vehicles
- ✓ 3-9% improvement documented in vehicle performance case studies by measuring power at the wheels
- ✓ TBO of piston cylinder group projected increase of 4 times

1. CASE STUDY OVERVIEW

Ideally the purpose of a lubricant is to provide a thin film that allows one surfaces to glide past another, without any direct contact. Under real world conditions, the contacting surfaces are not fully separated resulting in what tribologists call boundary lubrication. At this point in the operating regime, metal surfaces are in contact and need more than just lubricant to prevent excessive friction and detrimental wear[1].

A key benefit TriboTEX nanocoatings coatings provide, as they form on metallic surface interfaces, is a protective layer that prevents excessive wear. Through the process of development TriboTEX has explored the parameters of optimization for proprietary nano-sheet structures. Different dopant formulations have been developed to enhance nano-sheet particles for optimal performance over a versatile range of operation. TriboTEX formulations deliver unparalleled performance in unique environments and can be tuned to specialized applications. The following case study quantifies the level of wear protection characteristics nano-coating formulations exhibit in a bench test simulating boundary lubrication, with three points of contact.

ASTM Standard Test Method for Wear Preventive Characteristics of Lubricating Fluid (Four-Ball Method) D 4172

The American Society for Testing and Materials (ASTM) has developed a large series of long standing standards for assessing the performance of lubricant and petroleum products. The original Four-Ball Wear Test was introduced in 1964 (D 2266) for assessing the wear preventative characteristics of lubricating grease. In 1982 the ASTM issued a redesigned method for lubricants (D 4172), Test Method for Wear Preventive Characteristics of Lubricating Fluid[2]. This method has since become a widespread industry standard for testing the ability of lubricant formulations to prevent wear.

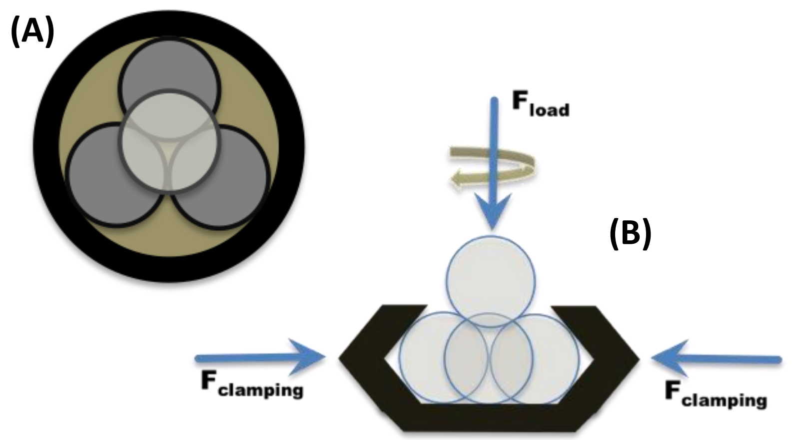

Figure 1. Schematic representation of the 4 Ball Wear Prevention Test. **(A)** Top view schematic **(B)** Side view schematic with arrows indicating specimen clamping force and test load.

Description of Methodology

This test method is often used to determine the relative wear preventive properties of lubricating fluids in sliding contact under a prescribed test load. The test specimen is constructed with three ½-inch diameter steel balls (Figure 1), clamped together and covered in the lubricant of interest. The lubricant inside the tray is maintained at a constant temperature (167°F), to maintain consistency across the tests. The fourth ball, attached to the loading arm, is pressed into the space at the center of the clamped three-ball specimen. The fourth ball, often called the top ball, is rotated at 1200 rpm under the test load for a specified time (60 min). The apparatus is then taken apart to inspect the resulting wear. Lubricant wear prevention characteristics are evaluated by measuring the diameter of the wear scar on each of the three test specimen balls. As recommended by ASTM standard, the case study was performed on a Multi-Specimen Friction and Wear Test Machine (Figure 2), manufactured by Falex Corp (Sugar Grove, IL). Wear volume was calculated as the volume of a calotte[3].

	Test Mode A	Test Mode B
Temp	167 ± 4°F (75 ± 2°C)	167 ± 4°F (75 ± 2°C)
Speed	1200 ± 60 rpm	1200 ± 60 rpm
Duration	60 ± 1 min	60 ± 1 min
Load	147 ± 2 N	392 ± 2 N

Table 1. Bench testing parameters for the Four-Ball Wear Test (ASTM D 4172)[4]

Figure 2. Bench test apparatus configured for the Four-Ball Wear Test. Industry standard for evaluating and optimizing the wear prevention characteristics of different lubricant formulations. The testing platform is a Falex Multi-Specimen Friction and Wear Test Machine.

A total of eleven different lubricant combinations were tested to better quantify the effects of varying lubricant formulations on wear prevention. Three baseline measurements were taken for comparison with two separate dopant optimization studies. The initial baseline was established using a neat mineral base oil to quantify wear under conditions without any additives. A second base line was performed by adding ground powder to base oil, to quantify how much wear protection synthesized nano-sheets provide. The third base line was performed using the base oil with TriboTEX synthetic nano-sheets. After the baseline measurements two optimization studies, using two different doping components (component A and component B), were performed. During the nano-sheet synthesis process two dopant components were introduced, at increasing concentrations, to different nano-sheet batches. The initial concentration of dopant components was 2.5% (*w/w*). Doping concentrations were doubled for each subsequent batch production synthesis resulting in three additional dopant concentrations for each component: 5%, 10%, and 20%. A total of eight lubricant conditions were generated, four for each component, and tested to measure wear

2. RESULTS

The Four-Ball Wear Test results clearly indicate the beneficial effects third body lubricants have on the wear protection characteristics of base oil (Table 2). Even finely ground powder provides substantially improved protection against wear when compared to base oil alone. The wear volume parameter indicates that ground powder addition results in a 10-fold wear protection improvement over base oil. Nano-sheets synthesized using the proprietary TriboTEX production process exhibit further improvements in performance against wear. Pure synthetic nano-materials exhibited improvement in wear protection characteristics when compared to ground powder, with a 4-fold decrease in the volume of worn material. These results demonstrate the ability of synthesized nano-sheets to drastically improve the wear protection characteristics of a base lubricant. The **volumetric wear rate in the specimens lubricated with base oil plus pure synthetic nano-sheets produced by TriboTeX is almost 40 times smaller** than in those lubricated by base oil alone.

	Base Oil	Ground Powder	Pure Synthetic
Wear Scar [µm]	786 ± 4.7	444 ± 3.5	315 ± 5.5
Wear Volume [µm^3] x 10^4	296 ± 7.28	29.96 ± 0.96	7.58 ± 0.54

Table 2. Wear parameters obtained from Four-Ball Wear Test.

Wear parameter results obtained from both dopant studies (Tables 3 and 4) highlight the necessity for tailored dopant selection and careful optimization of dopant concentration. Wear volume results from nano-sheets doped with a 2.5% concentration of Component A exhibited wear protection characteristics similar those of ground powder. Wear protection characteristics for this dopant package do not improve, upon those exhibited by pure synthetic nanao-sheets, until a 10% concentration is reached. Beyond the 10% concentration, at 20%, wear protecting characteristics of the lubricant start to decline below those of pure synthetic nano-sheets.

Synthetic nano-sheets w/	2.5% Dopant A	5% Dopant A	10% Dopant A	20% Dopant A
Wear Scar [µm]	416 ± 10.1	330 ± 23.2	318 ± 11.1	354 ± 22.2
Wear Volume [μm^3] x 10^4	23.2 ± 2.33	9.24 ± 2.88	7.94 ± 1.16	12.2 ± 1.35

Table 3. Wear parameters obtained from Four-Ball Wear Test using synthesized nano-sheets doped with varying concentrations of Component A.

Test results from the alternate dopant composition, Component B, exhibit similar behaviors to Component A. The deterioration in wear protection characteristics, however, does not decrease as drastically at low concentrations. The data indicate that the minimum wear volume is attained when Component B doping concentrations are approximately 10%. At this optimal concentration, doping synthetic nano-sheets with Component B improves the wear protecting characteristics of the lubricant. The data show a 14% decrease in wear volumes from those observed in pure synthetic nano-sheets.

Synthetic nano-sheets w/	2.5% Dopant B	5% Dopant B	10% Dopant B	20% Dopant B
Wear Scar [µm]	317 ± 7.5	316 ± 7.9	303 ± 6.2	316 ± 6.9
Wear Volume [μm^3] x 10^4	7.84 ± 0.77	7.76 ± 0.80	6.52 ± 0.55	7.71 ± 0.70

Table 4. Wear parameters obtained from Four-Ball Wear Test using synthesized nano-sheets doped with varying concentrations of Component B.

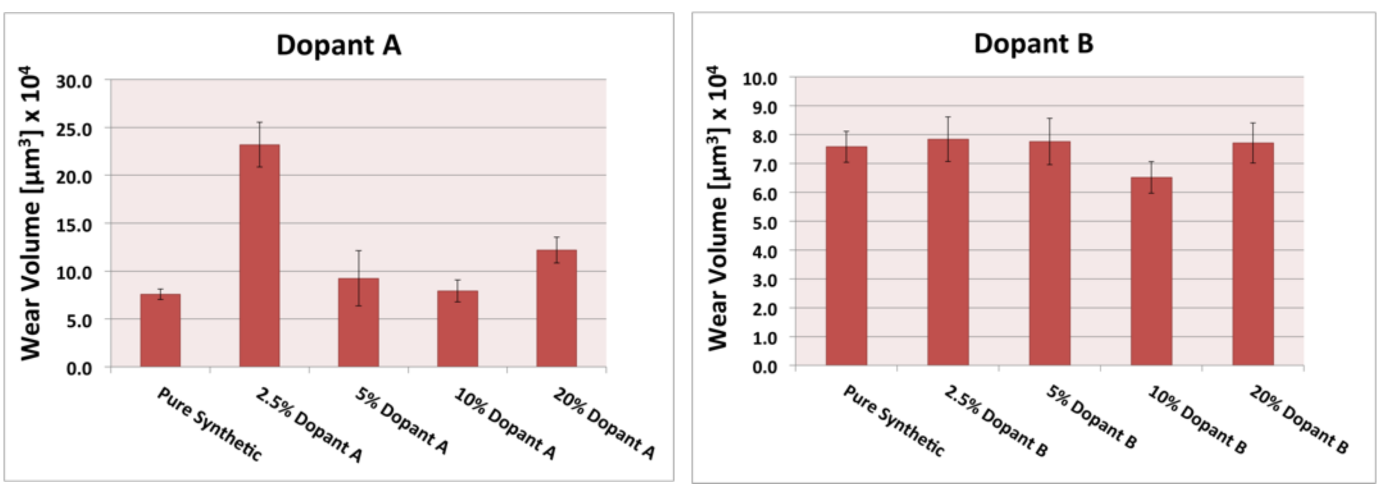

Figure 3. Wear volumes of synthesized nano-sheets doped with varying concentrations of Component A (left) and Component B (right) compared to pure synthetic nano-sheets measured with the Four-Ball Wear Method.

3. DISCUSSION OF FINDINGS

The results of this case study clearly illustrate the **enhanced wear protection characteristics** TriboTEX **nano-sheets provide** in base oil formulations. **Nearly 40 times less wear volume was observed in specimens with** TriboTEX **nano-sheets added** to base oil. The tests using varying dopant amounts indicate that doping synthetic nano-sheets does no produce a straightforward correlation to improved protection from wear. However, there are optimal concentrations of dopant formulations that produce more favorable outcomes. These case studies in wear protection characteristics highlights the benefits of finely tuned nano-material parameters with respect to wear protecting performance.

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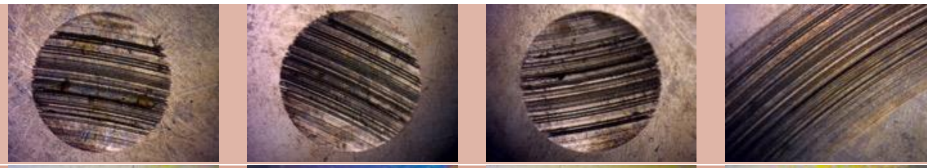
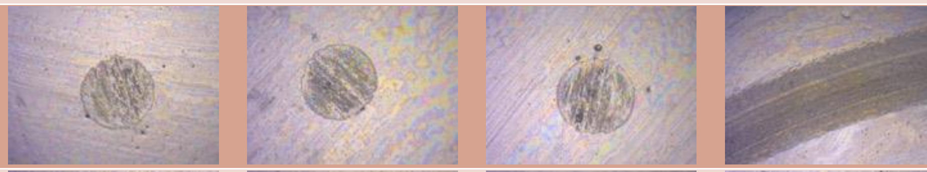
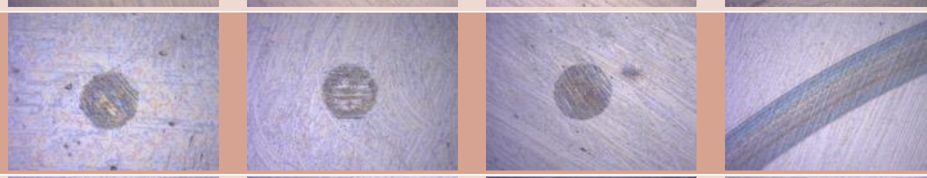
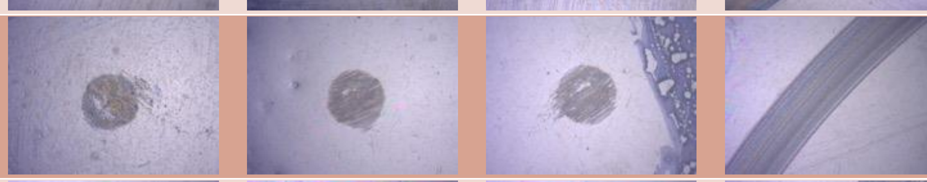
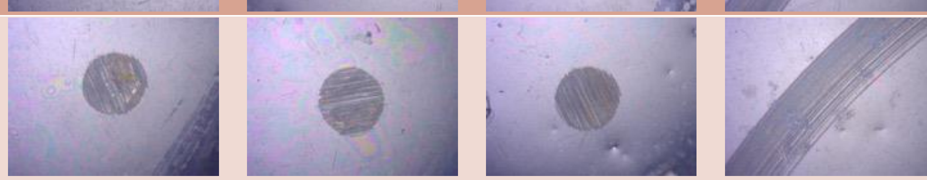
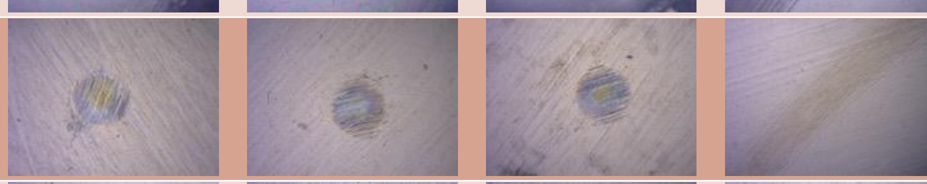
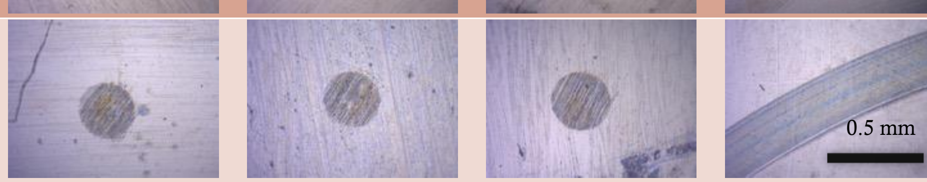
Appendix. 4-ball wear scar morphology test (ASTM D4172)

Sample #a0 Base Oil (reference) **Scar Diam** 786 ± 4.7 µm **Vol** 296 ± 7.28 x10^4 µm^3	
Sample #0 Ground Powder **Scar Diam** 444 ± 3.5 µm **Vol** 29.96 ± .96 x10^4 µm^3	
Sample #1 Pure Synthetic nano-sheets (no dopants) **Scar Diam** 315 ± 5.5 µm **Vol** 7.58 ± 0.54 x10^4 µm^3	
Sample #2 Dopant A 2.5% doped synthetic nanoparticles **Scar Diam** 416 ± 1 µm **Vol** 23.2 ± 2.33 x10^4 µm^3	
Sample #3 Dopant A 5% doped synthetic nanoparticles **Scar Diam** 330 ± 23 µm **Vol** 9.24 ± 2.88 x10^4 µm^3	
Sample #4 Dopant A 10% doped synthetic nanoparticles **Scar Diam** 318 ± 11 µm **Vol** 7.94 ± 1.16 x10^4 µm^3	
Sample #5 Dopant A 20% doped synthetic nanoparticles **Scar** 354 ± 22.2 µm **Vol** 12.2 ± 1.35 x10^4 µm^3	
Sample #6 Dopant B 2.5% doped synthetic nanoparticles **Scar Diam** 317 ± 7.5 µm **Vol** 7.84 ± 0.77 x10^4 µm^3	
Sample #7 Dopant B 5% doped synthetic nanoparticles **Scar Diam** 316 ± 7.9 µm **Vol** 7.76 ± 0.80 x10^4 µm^3	
Sample #8 Dopant B 10% doped synthetic nanoparticles **Scar Diam** 303 ± 6.2 µm **Vol** 6.52 ± 0.55 x10^4 µm^3	
Sample #9 Dopant B 20% doped synthetic nanoparticles **Scar Diam** 316 ± 6.9 µm **Vol** 7.71 ± 0.70 x10^4 µm^3	

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