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

Friction is the parasitic force that manifests at interface contacts in mechanical systems. The amount of friction and the determent of its presence depend on two main factors: the magnitude of the normal load and the interactions at the surface interfaces. Changing the normal load inside a mechanism is not an easy task once it has been designed, built, or is already in field. Costly redesigns before production often cannot be aimed at affecting the normal forces because of geometrical or functional constraints. This leaves surface modification as the only realistic alternative to reducing friction and detrimental wear in mechanisms of tomorrow. Many innovative and **promising solutions have been presented over the years but they are rarely cheap**. Diamond-like carbon (DLC) coatings have been slowly making their way from racetracks into production vehicles, at least for valve stems. Methods for applying plasma sprayed coatings in production have not yet reached their full potential and do nothing for existing vehicles, equipment, or machinery in use today.

TriboTEX, LLC has been synthesizing nano-sheets with functionally different sides (sticky/slick) for the purpose of coating metal surfaces during the course of operations. When added to base oils these nano-sheets travel through the lubricating system with ease and remain relatively inert. However at surface interfaces, experiencing a normal load, these nano-sheets are attached to metal surfaces from the pressure, exhibited by the normal load, and heat, supplied by friction. TriboTEX developed multiple optimized iterations of these nano-sheets by functionalizing the slick side of sheets with catalysts. Catalytic compounds further improve performance by seeding a lattice structure to promote DLC formation on the very top. These coatings could provide a novel approach to addressing the issues of friction, engine wear, and fuel consumption

Consumers testing prototype formulations in commuter vehicles have responded positively. Commonly reporting improvements in: power, fuel efficiency, and smoother engine operation. Consumer feedback provides an important metric of effectiveness under conditions encountered daily during real world driving. However, this feedback does not quantify nor demonstrate reductions in friction that nano-sheet coatings have been claimed to exhibit. This case study was performed to compare two lubricating oils under the same conditions to determine whether the claims of reduced friction are substantiated in bench tests with carefully controlled conditions.

ASTM Standard Test Method for Ranking Resistance of Materials to Sliding Wear Using Block-on-Ring Wear Test (G 77)

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 Method for Ranking Resistance of Materials to Sliding Wear Using Block-on-Ring Wear Test (G 77) is widely used in many industries to quantify friction in operational conditions. The elegant design of the Block-on-Ring Testing Method provides a direct measurement of sliding resistance (aka friction force). One of the most important attributes of this test is its simplicity and flexibility[1]. The test simulates a sliding interaction between two lubricated surfaces under a normal load and can be modified to approximate a variety of engine conditions and components. Commonly this bench test is employed to better understand friction in: camshafts, crankshafts, piston pins, connecting rods, and bearings[2,3].

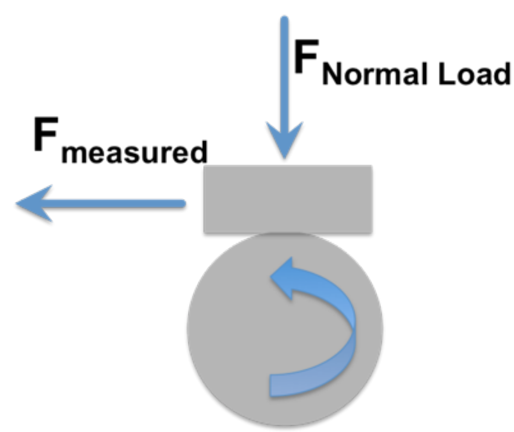

Figure 1. Schematic representation of the Block-on-Ring Test. Sliding resistance at the interface between the rotating ring and the stationary block are recorded with a load cell ($\mathbf{F_{measured}}$).

Description of Methodology

The case study was performed using a Falex Friction and Wear Testing Machine (model Falex-1, Falex Corp, Sugar Grove, IL). The testing apparatus (Figure 2) was outfitted with two thermocouples measuring temperatures of the lubricant bath chamber and the block test specimen (Figure 3). The original, manufacturer supplied 100-pound load cell (Western Load Cell Company, S1W) was used to measure the force produced by resistance to sliding at the interface. The signal was conditioned using an Interface Advanced Force Measurement transducer (M1029, Interface, Scottsdale, AZ). Measured temperature and force data were recorded to a laptop computer using a USB Data Acquisition System (DAQ series 55, Measurement Computing, Norton, MA) with the original logging software provided. The recommended calibration procedures described in Test Method for Calibration and Operation of the Falex Block-on-Ring Friction and Wear Testing Machine (ASTM D2714)[4] were used to ensure good mechanical operation of the test equipment.

Figure 2. Falex (model Falex-1) Block-on-Ring Friction and Wear Testing machine.

	Baseline	TriboTEX nano-sheets
Lubricant Composition	PAO 10	PAO 10 w/ TriboTEX
Speed	300 rpm	300 rpm
Duration	~20 hr	~20 hr
Load	300 N	300N

Table 1. Parameters used in ASTM G77 Block-on-Ring case study to measure friction coefficients at a sliding interface.

Case study tests were conducted using block and ring specimens supplied by Falex Corp. (part number H-60 and F-S10, respectively). After the cleaning and specimen placement procedures, in accordance to ASTM G77, the lubricant bath chamber was filled polyalphaolefin synthetic base stock (PAO 10), to establish a baseline measurement of the friction force.

The machine was turned on and allowed to reach the operational speed (300 rpm) selected for the test. Once a steady operational speed was reached the normal test load was applied (300N). After approximately 10 hours of testing at steady state conditions the normal load was decreased in a stepwise fashion from 300N to 50N, using 50N increments. This test modification was performed to probe performance in different lubricating regimes. Instrumentation set up and data collection were conducted over a 24-hour test period. At the end of the test cycle duration, the lubricant was drained from the chamber and the parts were cleaned. The same testing procedures were repeated for a second time using base oil with TriboTEX nano-sheet coatings.

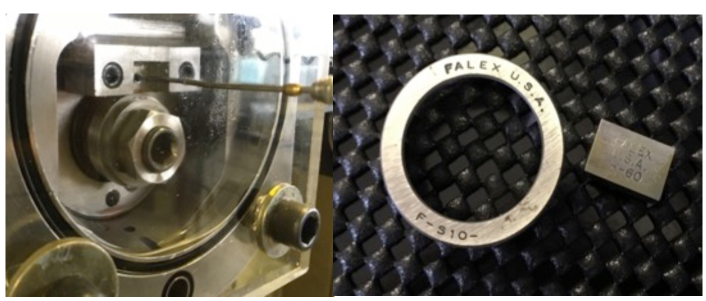

Figure 3. Thermocouple placed to measure block temperature (left). Manufacturer supplied block and ring test specimens (right).

2. RESULTS

The measured friction forces were plotted (Figure 4) without signal filtering to observe the full range of oscillations. The test cycle with Baseline oil performed, as expected. Like most good synthetic base oils, friction forces were from the start bellow 30N (coefficient of friction <0.1). As testing progressed beyond 5 hours (20,000s) the initial run-in period established a working surface interface and frictional resistance stabilized near 22.5N. The friction force for the baseline oil with TriboTEX nano-sheets, increased rapidly then seemed to remain near the same quantity as base oil. At approximately 4 hours of operation the friction force in the lubricant with TriboTEX nano-sheets underwent an extreme transformation in behavior; increasing above values of the PAO 10 baseline, then decreased rapidly below 1N. These rapid movements are indicative of surface property modification caused by material attachment at the surface. Once enough nano-sheets attached to the surface to influence the block-ring interface, friction declined dramatically. At the end of 10-hour into the test cycle PAO oil with TriboTEX nano-sheets **demonstrated a consistent and significantly lower friction force, measured at the sliding interface**.

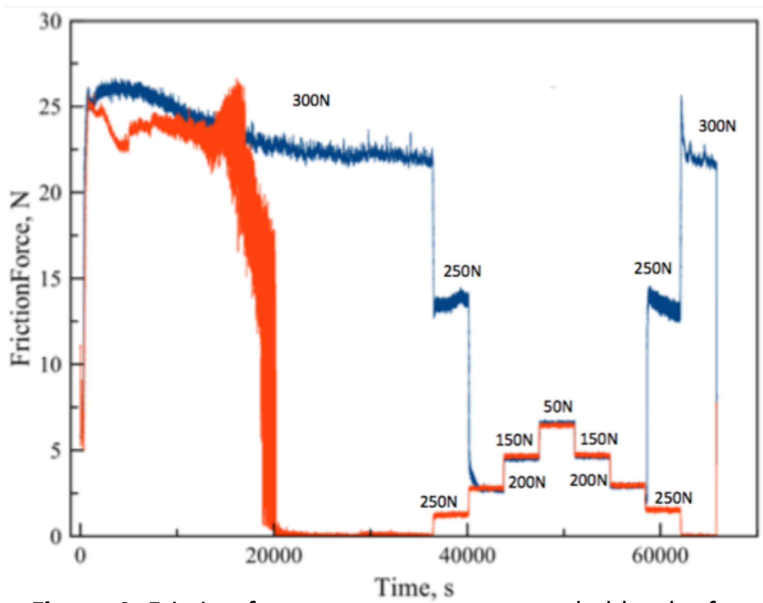

Figure 4. Friction force measurements recorded by the force transducer. Baseline (PAO 10) oil is indicated with the blue line. Baseline oil with TriboTEX nano-sheets is plotted with the red line.

3. DISCUSSION OF FINDINGS

Investigations of lubrication regimes at the interface were facilitated with step-wise decreases in the normal contact force. As expected, when the normal load in base oil force was initially reduced (250N) the friction force decreased dramatically, moving the interface from the boundary lubrication regime to a mixed lubrication regime. The next step reduction in normal loading (200N) reduces the friction force to its minimum, indicating that the interface is working in the elastohydrodynamic regime. The elsatohydrodynamic regime occurs when a thin film of lubricant separates the sliding surfaces, providing maximum protection to the interface. As the normal force decreases further (200N - 50N) the interactions at the interface enter the hydrodynamic regime, where the shear resistance of the oil separating the two surfaces determines friction. As the normal force is sequentially decreased the oil film between surfaces grows larger, increasing sliding resistance caused by the viscous drag. The same behavioral transformation of the lubricating regimes is observed, in PAO 10 base oil, as the instrument is loaded in a stepwise fashion. The oil film thins out until the optimal thin film conditions are reached, where minimal friction and wear occur (200N). Then as the load increases the lubricating regime at the interface moves to the mixed (250N) and the to the boundary (300N) lubrication regimes.

Behavior in the lubricant with the TriboTEX nano-sheet coating is quite different from the PAO 10 base oil. After the coating forms frictional resistance stabilizes to below 1N and remains steady. These results indicate that the coating allows the interface to support more load in the elastohydrodynamic lubrication regime. As the normal load is decreased a familiar pattern describing behaviors of interfaces in the hydrodynamic regime appears. The thicker oil wedge at the interface increases with it increasing drag. **The coated surface demonstrated an ability to carry larger a normal load while still maintaining the elastohydrodynamic regime at the interface**.

The results demonstrate the same phenomenon that is time and again observed in the field, when TriboTEX nano-sheets are tested in vehicle platforms. Specifically, that coating formation from individual nano-sheets is a relatively slow process. However, **once the nano-sheets are activated and attachment mechanisms begin the coating drastically reduces friction at the sliding interface**. The initial formation process can be clearly identified in the first 4 hours of the test. Once that initial formation process begins friction at the surface interface declines rapidly due to changes in surface properties. **The DLC coating allows the contacting surfaces to retain lubricity and transition to the elastohydrodynamic regime at a much higher load compared with quality synthetic base oil.**

REFERENCES

1. ASTM. Standard Test Method for Ranking Resistance of Materials to Sliding Wear Using Block-on-Ring Wear Test. *ASTM Int.* **G77-5,** 1–11 (2010).
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