Tribochemistry and Tribological Performance of Advanced Bearing Grease Fortified with Novel Self-Assembled Nanocarbon Additives

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COVID Positive

……..Only science can find a treatment for COVID-19
Objective

- Develop high performance grease with novel self-assembled nanocarbon additives for reliable and efficient lubrication and protection of rolling-element bearings
- Validate tribological performance of the grease formulations using bench testing related to the operating conditions/challenges of rolling-element bearings
- Investigate tribochemical reactions occurring at friction interfaces to correlate such mechanisms tribological properties

**Presentation Agenda**

- Introduction & Tribological Challenges of Rolling Bearings
- Brief Description of Self-Assembled Nanocarbon Additive Technology
- Description of Test Lubricants and Test Methods
- Bench Test Evaluation of Tribological Performance
- Tribochemical Analysis of Friction Surfaces

- FE8 Wear Test
- Fretting Wear
- HFRR
- Block-on-Ring
- 4-Ball EP & Wear
Rolling Bearings

Rolling bearings are used in all main shaft and auxiliary drive shaft applications to support and guide rotating or oscillating elements and transfer loads.

**Single-Direction Thrust Ball Bearing**

**Single-Row Deep Groove Ball Bearing**

**Tapered Roller Bearing**

**Spherical Roller Bearing**

**Single-Row Angular Contact Ball Bearing**

**Cylindrical Roller Bearing**

Mining, petroleum production, wind turbines, power generation, power transmission, cement processing, aggregate crushing, and metal recycling, Briquetting machines, rubber mixing equipment, rolling mills, rotary dryers, or pulp and paper machinery, construction equipment, crushers, electric motors, blowers and fans, gears and drives, plastics machinery, machine tools and traction motors and pumps and many more….
Lubrication in Rolling Bearings

Avoid or reduce metal-to-metal contact between rolling and sliding contacts

Mostly Grease Lubrication

Tribowear of Rolling-Element Bearing

Adhesive Wear
Abrasive Wear
Fretting Wear
False Brinelling

The life of rolling bearings is influenced by the lubricant film

Hydrodynamic lubrication
Mixed lubrication
Boundary lubrication

The life of rolling bearings is influenced by the lubricant film

Base Oil
Thickener System
Lubricant Additives
EP, AW & Friction Modifier
Carbon Nanomaterials: Boundless Possibility

High electron mobility;
High thermal conductivity;
High current density;
Transparent and conductive properties;
Metallic/semiconductive properties

Electronics applications such as:
Transistors,
Transparent thin films,
LSI wiring etc.

Nano-scale structures;
Ion adsorption;
High surface area;
Catalysis support

Energy storage applications such as:
Fuel cell, capacitor,
Lithium ion battery etc.

Bio-technology applications such as:
Drug delivery system,
Bio sensor,
Cell cultivating etc.

High surface area; Strong adsorption; High affinity binding

Nanostructures;
Slim and strong;
Light weight; High thermal conductivity;
High physical strength;
High electronic conductivity;
Metallic/semiconductive properties

Nanotechnology e.g. Aerospace, Defence, automotive etc.

[Courtesy: https://edu.rsc.org/infographics/allotropes-of-carbon/4012885.article]
Nanocarbon Additives: Self-Assembled Carbon Nanoarchitectonics

Patent-pending mechanochemical-assisted nanoarchitectonics approach of concurrent top-down & bottom-up fabrication to generate nanolubricant additives with a variety of organic self-assembly with carbon nanostructures

Applications
- Engine Oils
- ATF
- Greases
- Gear Oils
- Industrial Lubricants
- Metal Forming Lubricants
Nanocarbon Additives: Self-Assembled Carbon Nanoarchitectonics

Patent-pending technology can generate organofunctional monolayers ranging from 2-5 nm for reactivity with a wide range of organic compounds to form self-assembled nanostructures.
Nanocarbon Additives: Self-Assembled Carbon Nanoarchitectonics

Unipolar or Multispecies Inorganic sp² and/or sp³ Hybridized Nanocarbon

Surface-Triggered Reactions to Form Reactive Organofunctional Groups

X-ray Diffraction Patterns of Example Organofunctionalized sp² and sp³ Nanocarbon
# Test Materials

## Grease Lubricants

<table>
<thead>
<tr>
<th></th>
<th>Comp A</th>
<th>Comp B</th>
</tr>
</thead>
<tbody>
<tr>
<td>NLGI Grade</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Thickener</td>
<td>Lithium-Complex (LiX)</td>
<td>Calcium Sulfonate Complex (CaSX)</td>
</tr>
<tr>
<td>Base Oil</td>
<td>Synthetic, PAO</td>
<td>Synthetic, PAO</td>
</tr>
<tr>
<td>Base Oil Viscosity, 40° C</td>
<td>220 cST</td>
<td>198 cST</td>
</tr>
<tr>
<td>Worked Penetration, 60 strokes, 77 °F (25 °C)</td>
<td>320</td>
<td>319</td>
</tr>
<tr>
<td>Worked Penetration, 10,000 strokes, 77 °F (25 °C)</td>
<td>328</td>
<td>329</td>
</tr>
<tr>
<td>Dropping Point, °F (°C)</td>
<td>520 (271)</td>
<td>650 (343)</td>
</tr>
</tbody>
</table>

## Additives

<table>
<thead>
<tr>
<th></th>
<th>Additive A (A1)</th>
<th>Additive B (B1)</th>
<th>Additive C (C1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nanocarbon Type</td>
<td>Graphene Oxide</td>
<td>Graphene Oxide</td>
<td>Nanodiamond</td>
</tr>
<tr>
<td>Particle size</td>
<td>0.5-1.5 µm</td>
<td>0.5-1.5 µm</td>
<td>4-5 nm</td>
</tr>
<tr>
<td>Graphene thickness</td>
<td>&lt; 5 nm</td>
<td>&lt; 5 nm</td>
<td></td>
</tr>
<tr>
<td>Organofunctional</td>
<td>Amine;</td>
<td>Amine;</td>
<td>Amine;</td>
</tr>
<tr>
<td>Compounds</td>
<td>Organo Molybdenum Compounds</td>
<td>Heterocyclic Compounds</td>
<td>Organo Molybdenum-Heterocyclic Hybrid</td>
</tr>
</tbody>
</table>
# Test Methods

<table>
<thead>
<tr>
<th>Test Methods</th>
<th>Operating Conditions</th>
<th>Critical Grease Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>FE 8 (DIN 51819) Wear Test</td>
<td>Stop/start and low speed</td>
<td>Antiwear behavior</td>
</tr>
<tr>
<td>4-Ball Wear Test (ASTM D2266)</td>
<td></td>
<td>Energy efficiency from low friction</td>
</tr>
<tr>
<td>Block-on-Ring Tribometer</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4-Ball EP (ASTM D2596)</td>
<td>Rapid changing loads</td>
<td>Extreme Pressure Load Carrying Capacity</td>
</tr>
<tr>
<td>High Frequency Reciprocating Rig (HFRR)</td>
<td>High thrust movement</td>
<td>Wear protection, Film strength</td>
</tr>
<tr>
<td>Fretting Wear (ASTM D4170)</td>
<td>Vibrations/oscillation</td>
<td>Resistance against fretting wear &amp; corrosion</td>
</tr>
<tr>
<td>SRV Fretting Wear (ASTM D7594)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

## Microanalysis of Friction Surfaces

- Tribofilm Morphology and Analysis: Scanning Electron Microscopy and Energy Dispersive X-Ray Spectroscopy
- Tribochemical Analysis: Raman Spectroscopy

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FE8 (DIN 51819) Wear Test
Determination of Wear on Rolling Components

31312 Tapered roller bearing
Grease quantity ~ 200 g
Temperature- Ambient

S1: Testing in progress

<table>
<thead>
<tr>
<th>Stages (S)</th>
<th>Axial Load (KN)</th>
<th>Speed (RPM)</th>
<th>Duration</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1: Boundary Lubrication: High load &amp; low speed</td>
<td>80</td>
<td>7.5</td>
<td>500 hours</td>
</tr>
<tr>
<td>S2: Mixed Lubrication: Medium load &amp; speed</td>
<td>50</td>
<td>75</td>
<td>500 hours</td>
</tr>
<tr>
<td>S3: EHL : Low load, high speed</td>
<td>20</td>
<td>1500</td>
<td>500 hours</td>
</tr>
</tbody>
</table>

S1: MW50 (ROLLERS), MG

S2: MK50 (CAGE), MG

S3: MW50 (ROLLERS), MG

MK50 (CAGE), MG

Comp A  Comp A+A1  Comp A+B1
Four Ball EP Test (ASTM D2596)
Determination of Extreme Pressure Load Carrying Capacity

Grease quantity - 3 g
Time – 10 seconds
RPM - 1760
Load – 0-1000 KGF
Temperature – 80 ± 15 °F
Test Specimen – Falex- 0.5 in. Diameter, AISI E-52100 Grade 25, Rc 64-66 (Extra Polish)
Four Ball Wear Test (ASTM D2266)
Determination of Adhesive Wear & Friction

- Grease quantity - 3 grams
- Time - 60 Minutes
- RPM - 1200
- Load - 40 KGF
- Temperature – 75 °C
- Test Specimen – Falex- 0.5 in. Diameter, AISI E-52100 Grade 25, Rc 64-66 (Extra Polish)
Block-On-Ring Tribotest
Determination of Sliding Friction Behavior

- Load - 33 KG
- Speed - 500 RPM
- Test Duration - 30 minutes
- Lubricant - 80 ml
- Test Specimen - 52100 H/P Block (Falex)
- Ring - Timken A4138

Block-on-Ring Tribotest were performed on Synthetic PAO Base Oils - ISO 220 and ISO 150

ISO 220: Coefficient of Friction Measurements

![Graph showing coefficient of friction measurements for ISO 220 with different lubricants: As received GO, + C1, + B1, + A1.](image)

- COF_{Average} = 0.1
- COF_{Average} = 0.056
- COF_{Average} = 0.041
- COF_{Average} = 0.036

Precipitate Settling in PAO 220
Block-On-Ring Tribotest
Determination of Sliding Friction Behavior

Load: 33 KG, Speed: 500 RPM, Test Duration: 30 minutes, Lubricant: 80 ml

ISO 150: Coefficient of Friction Measurements
- COF Average: 0.19
- COF Average: 0.158
- COF Average: 0.09
- COF Average: 0.072

ISO 150: Temperature Measurements
Thermal Conductivity & Electrical Conductivity Correlation

Naphthenic Oil

% Enhancement in Thermal Conductivity

Dielectric breakdown voltage (kV)

% ENHANCEMENT - THERMAL CONDUCTIVITY

DIELECTRIC BREAKDOWN VOLTAGE (KV)
High-Frequency Reciprocating Rig (HFRR) Determination of Wear Resistance & Film Stability

Lubricant- 2 ml, Load- 200 g, Time- 75 min, Frequency- 50 Hz, Stroke- 1mm, Temperature- 140 °F (60 °C)

- **COMP A**
  - WSD$_{mean}$: 312 μm
  - COF$_{Average}$: 0.128
  - Film: 84%

- **COMP A + A1**
  - WSD$_{mean}$: 155 μm
  - COF$_{Average}$: 0.055
  - Film: 100%

- **COMP A + B1**
  - WSD$_{mean}$: 171 μm
  - COF$_{Average}$: 0.096
  - Film: 93%
High-Frequency Reciprocating Rig (HFRR) Determination of Wear Resistance & Film Stability

- **COMP B**
  - WSD: 297 µm
  - Film: 80%
  - COF<sub>Average</sub>: 0.123

- **COMP B + A1**
  - WSD: 159 µm
  - Film: 100%
  - COF<sub>Average</sub>: 0.07

- **COMP B + B1**
  - WSD: 169 µm
  - Film: 95%
  - COF<sub>Average</sub>: 0.093

- **COMP B + C1**
  - WSD: 139 µm
  - Film: 100%
  - COF<sub>Average</sub>: 0.059
Fretting Wear
Determination of Wear Resistance Under Vibrations/Oscillatory motion

Load - 2450 N, Frequency – 30 Hz., Test Duration- 22 hours, Lubricant- 1.0 ± 0.05 grams, Swing Angle- 12 °, Test Specimen – Thrust type bearings (Qty. 2)

![Graph showing wear scar diameter and bearing race mass loss](image-url)
Tribofilm Morphology and Chemical Analysis
SEM-EDS Microanalysis

COMP B + A1

Wear track

Smeared Film

Carbon Tribofilm

EDS Elemental Mapping

C-K

Cr-KA

Fe-KA

S-KA

Si-KA

Al-KA

Mo-LA

EDS Elemental Peak Analysis
Tribofilm Morphology and Chemical Analysis
SEM-EDS Microanalysis

COMP A + A1
Wear track
Smeared Film

Carbon Tribofilm
EDS Elemental Mapping

EDS Elemental Peak Analysis

C-KA
Cr-KA
Fe-KA
S-KA
Tribofilm Morphology and Chemical Analysis
SEM-EDS Microanalysis

COMP A + B1

EDS Elemental Mapping
Carbon Tribofilm

EDS Elemental Peak Analysis
C-KA
Cr-KA
Fe-KA
S-KA
Tribochemical Analysis
Raman Spectroscopy

D-peak – 1350 cm\(^{-1}\)
G-peak - 1580 cm\(^{-1}\)
2D-peak - 1580 cm\(^{-1}\)

Increased intensity ratio of D to G peak in tribofilm formed in wear scar and wear tracks indicating disordered graphitic structure in friction surfaces.
Tribochemical Analysis
Raman Spectroscopy

Tribofilm Similar to Graphitized DLC Transfer Layers/Films
Conclusions

• Tribological performance and tribochemical analysis of novel self-assembled nanocarbon additives were successfully evaluated in Lithium complex and Calcium sulphonate grease designed for rolling-element bearings.

• Three nanocarbon additives were tested consisting of graphene oxide organofunctionalized with amine, organomoly & organosulfur and nanodiamond organofunctionalized with amine and organomoly-organosulfur complexes. All nanocarbon additives showed high compatibility and stability in PAO base oils.

• Tribological performances were measured using FE8 wear tests, fretting wear, block-on-ring, 4-ball tests, and HFRR bench testing related to the operating conditions/challenges of rolling-element bearings.

• Grease formulations containing organosulfur-functionalized graphene demonstrated better extreme pressure (EP) load carrying property. In comparison, organomoly-functionalized graphene oxide showed better antiwear characteristics and friction modification.

• Hybrid organomoly-organosulfur functionalized nanodiamond additive was specially designed for a high-performance heavy-duty calcium sulphonate complex grease and it demonstrated excellent synergy and tribological performance in terms of high EP, antiwear, and friction modification.

• With graphene based nanoadditives, carbon tribofilms with disordered graphitic structure were formed at the friction interfaces.

• With nanodiamond based additives, tribofilms like graphitized DLC transfer layers were formed at the friction interfaces.
Acknowledgements

Thank You!