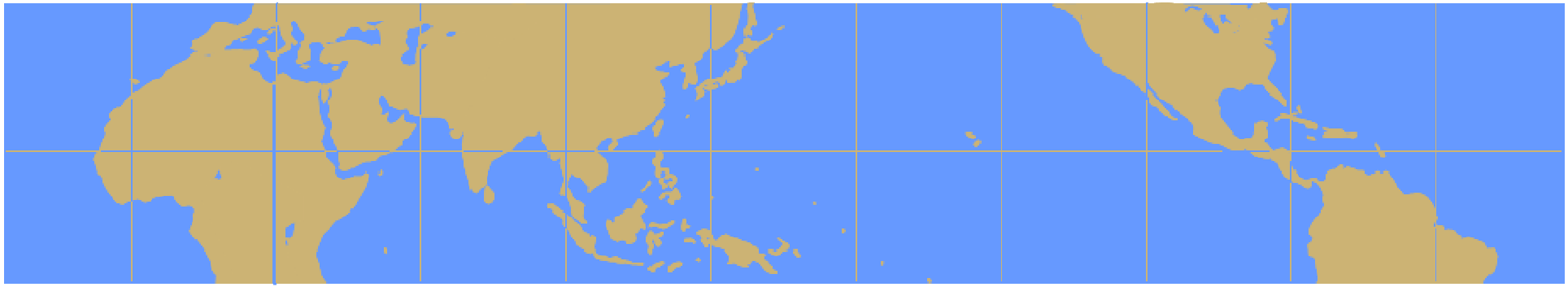


ASTM Subcommittee D02.G on Lubricating Grease

- Update on Activities

David Turner
Shell Global Solutions (US) Inc.

John Graham
Exxon Mobil Research & Engineering

Introduction to ASTM

- ASTM International
  - Formed in 1898, to address railroad steel quality specifications
  - Now one of the world's largest voluntary standards development organizations
- Mission statement
  - To be recognized globally as the premier developer & provider of voluntary consensus standards, related technical information, & services that:
    - promote public health & safety, support the protection & sustainability of the environment, & the overall quality of life
    - contribute to the reliability of materials, products, systems & services
    - facilitate international, regional, & national commerce
- Participation & membership is open to anyone

ASTM Overview

- Some basic statistics
  - Over 140 technical committees
    - Involved in a wide range of technical & management sectors
  - Over 31,000 members from more than 175 countries
  - Over 11,000 active standards used internationally
- Membership balanced across interests
  - Producers, users, consumers, general interest parties (e.g. academicians & government representatives)
- Objectives
  - To ensure the fair representation & participation of key stakeholders in ASTM International activities
  - To help assure the development of technically sound, market relevant standards.

Subcommittee D02.G

Scope:

- Lubricating greases & grease components
  - Base fluids, thickeners, & additives
- Promotion of technical knowledge
- Development & maintenance of
  - Standard test methods
  - Specifications, guides, practices
  - Terminology

Subcommittee D02.G

- Officers:
  - Chair — David Turner
  - Vice Chair — Raj Shah
  - Secretary (ballots) — Matt Sivik
  - Secretary (minutes) — John Graham
  - Secretary (membership) — John Sander

- ASTM Staff
  - Staff Manager — David Bradley
  - Administrative Assistant — Lisa Drennen
  - Editorial Assistant — Nicole Baldini

Subcommittee D02.G Structure

- Seven Sections
  - Section 1: Chemical & General Laboratory Tests – Joe Kaperick
  - Section 2: Consistency & Related Rheological Tests – Tom Boersig
  - Section 3: Physical Tests – Steve Humphreys
  - Section 4: Functional Tests – Tribology – Mike Anderson
  - Section 5: Functional Tests – Temperature – John Graham
  - Section 6: Functional Tests – Contamination – Matt Bailey
  - Section 7: Research Techniques – Gareth Fish

- Related Groups
  - D02.09.E: Oxidation of Lubricating Grease – John Graham
  - D02.B0.04: Automotive Greases – Gareth Fish
  - USA TAG Grease Panel – David Turner

Current Issues

- SI units initiative
  - ASTM is working to convert all test methods to SI units
- ASTM initiative to eliminate mercury from standards
  - Subcommittee G is working to implement this
  - Mostly affects mercury-in-glass thermometers
- Elimination of undesirable or obsolete solvents
  - Removal of chloroform & 1,1,1-trichloroethane
  - Replacement of D235 specification mineral spirits
- Standardization of equipment cleaning methods
  - Questionnaire distributed to full Sub G membership

Section 1 - Standards

- D128 Grease Analysis
- D1404 Deleterious Particles
- D4048 Copper Corrosion
- D4289 Elastomer Compatibility Practice
- D6185 Grease Compatibility Practice
- D7527 Antioxidant Contact by Linear Sweep Voltammetry (RULER Test)
- D7718 In-Service Grease Sampling Practice

D128 Grease Analysis

- Oldest lubricating grease standard
  - Originally published in 1922
  - Includes many basic definitions
  - Tests for thickener, base oil, free alkali, free fatty acid content, etc.
- Currently being heavily revised
  - Keep only sections relevant to contemporary greases & still in use
  - May be issued as separate new standards

D1404 Deleterious Particles

- Counts abrasive particles in grease
  - Related to grease cleanliness
- Plastic plates scratched by abrasive particles
  - Report includes pressure applied & number of scratches
- NLGI Technical Committee on grease cleanliness
  - NLGI-ELGI Working Group & mini round-robin
  - D1404 method under evaluation
    - Comparison with other methods (e.g. Hegman Gauge, FTM 3005.4, DIN 51 813)
    - Identify potential improvements to method

D7527 Antioxidant Content by Voltammetry

- Published in 2010
- Measures remaining primary antioxidants (phenolic & aminic) in the product
  - Estimation of remaining useful service life of the product
- Measurement performed using the RULER instrument
  - Established technology with proven reputation
  - Assist with recommendation for continued use or replacement of in-service grease

D7718 In-Service Grease Sampling Practice

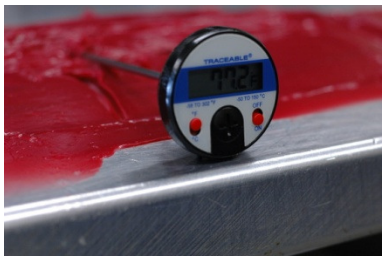
- Need identified for a standard guide for sampling in-service greases
  - Gearboxes, electric motors, actuators, etc
- In-service grease sampling more difficult than oil
  - How to obtain "representative" sample ?
  - Guide addresses multiple issues:
    - Location for sampling (proximity to contact surfaces)
    - How to extract sample (syringe, spatula, collector)
    - Maintaining sample integrity (during/after sampling)
    - Coping with inhomogeneous samples

Section 2 - Standards

- D217 Cone Penetration
- D1092 Apparent Viscosity
- D1403 Small-Scale Cone Penetration
- D1831 Roll Stability
- D7342 Shear Stability in the Presence of Water

D217 Cone Penetration

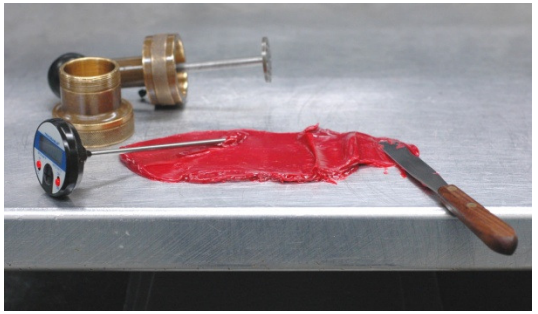
- Methods for undisturbed, unworked, worked, extended worked, & block penetration
  - Originally published in 1925
- ASTM definition of "lubricating grease"
  - Lists NLGI consistency grades



- Revised standard issued in 2010
  - Now specifies 3 measurements from a single specimen
    - Previously required 3 specimens for NLGI 3 & softer greases
  - Allows use of alternate sample cooling methods
  - Recent round-robin to confirm & update precision statements

D1403 Small-Scale Cone Penetration

- ½-scale & ¼-scale versions of D0217
  - Unworked & worked penetration only
  - Manual working
- Used when sample quantity is limited
- Used in some other test methods
  - e.g. D1831 Roll Stability
- Revised standard issued in 2010
  - Similar modifications to D0217
  - Recent round-robin to confirm & update precision statements



D7342 Shear Stability in the Presence of Water

- New standard added in 2007, reapproved in 2012
- Two procedures:
  - Wet working (100,000 strokes)
  - Wet roll stability (2 hours @ room temperature)
- 10% Water added to grease
  - Premixed into grease before test (worker)
  - Added separately at start of test (roll stability tester)
- Measure change in penetration
  - Shear stability in the presence of water

Section 3 - Standards

- D566 Dropping Point
- D972 Evaporation Loss
- D1742 Oil Separation
- D2265 Dropping Point (wide temp range)
- D2595 Evaporation Loss (wide temp range)
- D4425 Oil Separation (centrifugal)
- D6184 Oil Separation (conical sieve)

D566 Dropping Point

- Original dropping point test method using oil bath
  - First published in 1940
  - Limited to 288°C maximum temperature
- Balloted for withdrawal in 2008
  - 2 negative votes received
  - Still referenced & required by some users
    - e.g. Military grease specifications
- Standard reapproved in 2009

D2265 Dropping Point (wide temperature range)

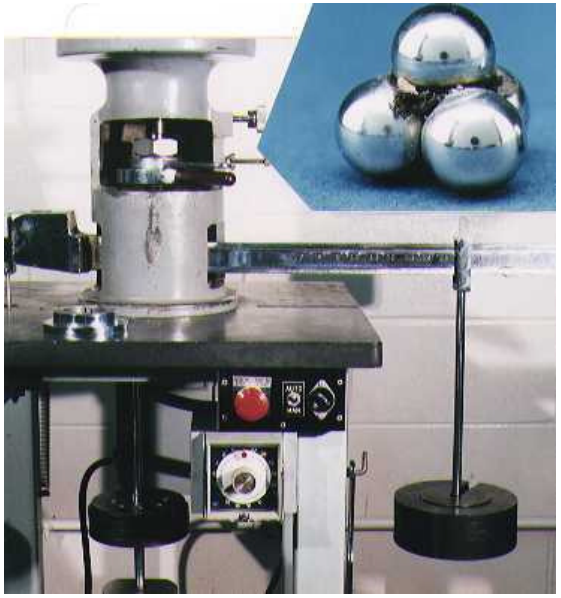
- Aluminum block test method
  - Wide temperature range
  - Up to 309°C maximum temperature
- Used for high temperature greases:
  - Complex soap, polyurea, calcium sulfonate thickeners
- Manual method using mercury thermometer
  - Difficult to find alternatives to mercury
  - Rate of heat transfer & response time critical
  - Candidate PRT system may be $3K/each for initial trials

Section 4 - Standards

- D2266 Four-Ball Wear
- D2509 Timken
- D2596 Four-Ball EP
- D4170 Fretting Wear (Fafnir)
- D5706 EP by SRV
- D5707 Friction & Wear by SRV
- D7420 Tribomechanical Properties of Grease Lubricated Plastic Socket Suspension Joints by SRV
- D7594 Fretting Wear Test by SRV

D2266 & D2596 Four-Ball Wear & EP

- Four balls in pyramid configuration
  - Three balls fixed, one ball spinning
- D2266 Wear Test
  - 1200 RPM, 75°C, 40 kg, 60 minutes
    - Measure scar diameter on fixed balls
- D2596 EP Test
  - 1770 RPM, 27°C, 10 seconds/load
    - Increasing load stages; run to weld point
  - Plan to harmonize conditions with oil test
  - US & European versions operate at different speeds



SRV-Based Standards

- D5706 (EP), D5707 (Friction & Wear), D7420 (Plastic Suspension Joints), D7594 (Fretting Wear)
  - All utilize ball-on-disk or pin-on-disk configuration
  - Can increase step load to lubricant film rupture
- Variables:
  - Temperature
  - Applied Load
  - Stroke length
  - Frequency of oscillation
  - Test piece materials

Section 5 - Standards

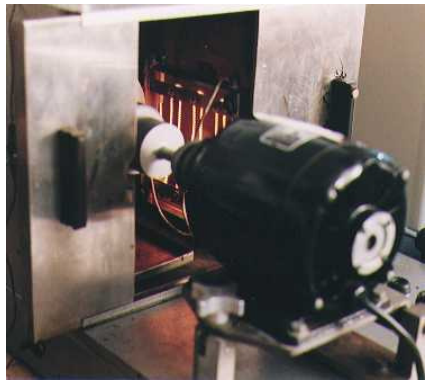
- D1263 Wheel Bearing Leakage
- D1478 Low-Temperature Ball Bearing Torque
- D3336 High-Temperature Ball Bearing Life
- D3527 Wheel Bearing Life
- D4290 Wheel Bearing Leakage (Accelerated)
- D4693 Low-Temperature Wheel Bearing Torque

D1263 Wheel Bearing Leakage

- 1930's Ford front wheel bearing hub
  - Conditions: 660 RPM, 105°C, 6 hours
  - Leakage reported in grams
- Balloted for withdrawal in 2010
  - Approved with no negatives
- Superseded by D4290 accelerated test

D3527 High Temperature Wheel Bearing Life

- Same hardware as for D4290 (Leakage) test
  - Based on 1950's front wheel hub assembly
    - 1000 rpm, 111 N thrust load, 160°C
    - Cyclic operation: 20 hours on, 4 hours off
  - Electric motor current measured
    - Failure based exceeding allowed increase
- D3527 test precision issues
  - Task force investigation
    - Identify inconsistencies, ambiguities
    - Evaluate potential effects of major changes
      - Reproducibility & test severity

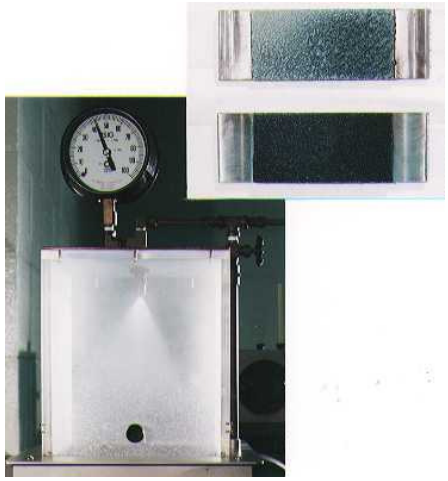


Section 6 - Standards

- D1264 Water Wash-Out
- D1743 Rust Test (Distilled Water)
- D4049 Water Spray-Off
- D5969 Rust Test (Synthetic Sea Water)
- D6138 Dynamic Rust Test (Emcor)

D4049 Water Spray-Off

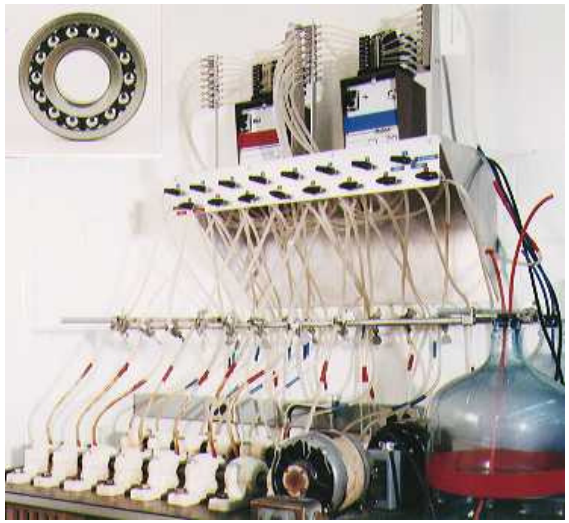
- Direct impingement of a water spray on a grease-coated steel plate
  - Grease film – 0.8 mm (1/32 inch)
    - Water at 38°C (100°F), 276 kPa (40 psi)
    - 5 minutes spray time
  - Results reported as % loss



- Mini round-robin conducted
  - Clarify specification & designation for spray nozzle
    - Orientation, calibration, cleaning

D6138 Dynamic Rust Test (Emcor)

- SKF test method
  - 1306 K double-row self-aligning ball bearings (steel cage)
  - Two bearings per grease sample (i.e. duplicate)
  - 168 hour test cycle:
    - 3 x (8 hours on, 16 hours off)
    - Then 108 hours off
  - Distilled water, or corrosive solution
- Rating scale:
  - From '0' (no corrosion)
  - To '5' (>10% corrosion)

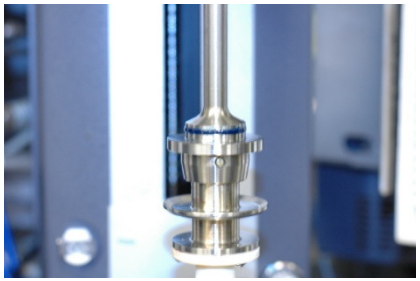


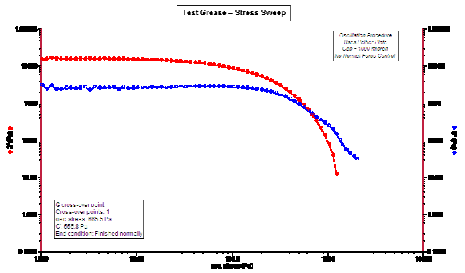
Section 7 – Test Method Development

Main activities:

- Grease Rheometry
- Lincoln Ventmeter Test Standardization
- Grease Density Measurement
- Thin Film Thermal Stability

Grease Rheometry





- Potential to be powerful technique
  - Useful for evaluation of in-service greases
    - Small sample size (~5 g)
  - Test under controlled conditions
    - e.g. applied stress, temperature, frequency
- Initial focus on a fundamental test
  - Controlled stress-sweep
    - Irrecoverable breakdown of thickener structure
- More complex methods to follow

Lincoln Ventmeter Test Standardization

- Grease mobility at low temperatures
- Standardization of well-known Lincoln test method
  - Small-scale version of centralized distribution system
    - 762 cm (300 in) of 3 mm (⅛-in) tubing
- Grease pressurized at start of test
  - Measures time required for the pressure to drop to a predetermined value
  - Establish recommended minimum operating temperature

D02.09.E Grease Oxidation - Standards

- D942 Pressure Vessel Oxidation
- D5483 Oxidation Induction Time by Pressure Differential Scanning Calorimetry

D5483 PDSC Oxidation Induction Time

- Static oxidation test
  - Very small sample size (2 mg)
  - 2.5 MPa (500 psi) pure oxygen
  - Temperature 155–210°C
- Exotherm on chart indicates oxidation
  - Oxidation induction time reported in minutes
    - Result at highest temperature with induction time ≥10 minutes
- Current standard developed on specific manufacturer's equipment
  - Round-robin planned to establish updated precision statement using equipment from multiple manufacturers

D02.B0.04 Automotive Greases - Standard

- D4950 Standard Classification & Specification for Automotive Service Greases
  - Developed by SAE, ASTM, & NLGI
  - Defines wheel bearing grease categories GA, GB, & GC
  - Defines chassis grease categories LA & LB
- NLGI policy:
  - Only GC & LB service marks may be displayed

USA Technical Advisory Group Grease Panel

- Technical Advisory Group to ANSI (American National Standards Institute)
  - ANSI is US voting member of ISO
- TAG Provides input & voting advice to ANSI for ISO standards related to lubricating grease
- Some ASTM D02.G Section Chairs also chair ISO working groups

Areas of Possible Future Interest

- Grease Noise Testing
  - e.g. SKF BeQuiet+, FAG MGG
- Flammability Resistance
  - Of major interest for Steel Mills
- Additional Rheology methods
  - More complex evaluations
- Automatic Dropping Point
- Micro-oxidation (Penn State)

vs

Other ASTM Activities

- Inter-Laboratory Correlation Program (ILCP)
  - 41 proficiency programs covering variety of products
  - Grease program runs in April & October
    - Participating labs receive coded reports with statistical analysis
    - Can form part of lab Quality System processes
- ASTM Certification Programs
  - New initiative by ASTM
  - By request, may add:
    - Personnel certification program
    - Product certification program

Feedback Request

- Please advise David Turner (david.turner@shell.com), or any subcommittee officer, of any grease test method development needs
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Questions?

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